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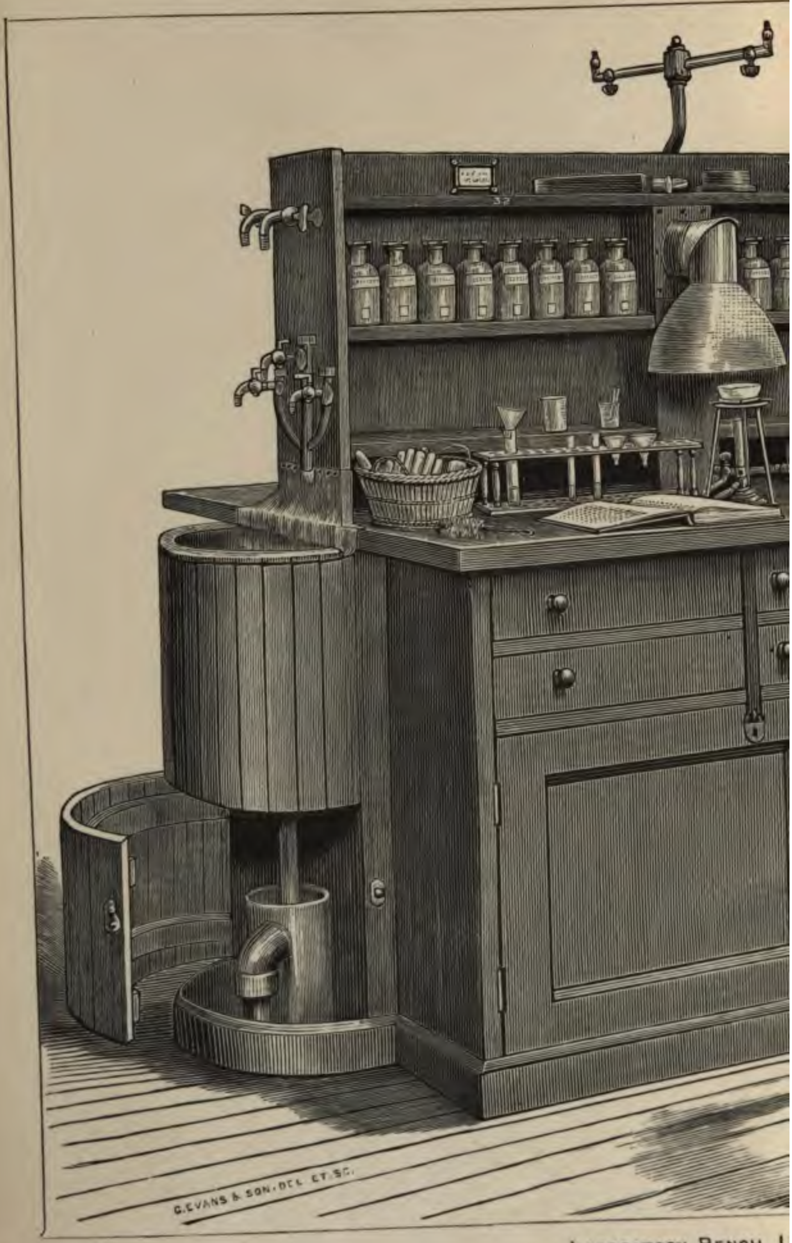
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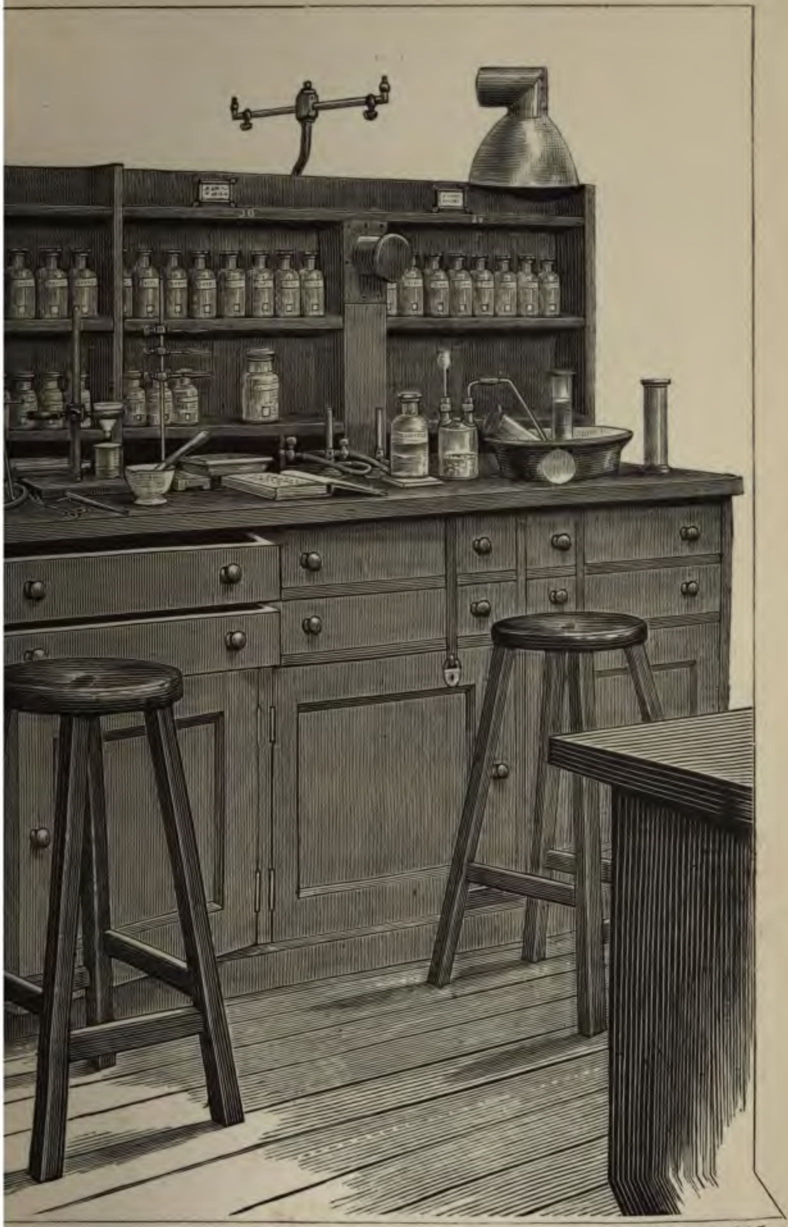

COLLEGE, NOTTINGHAM.

See page 315.

A TREATISE

ON

PRACTICAL CHEMISTRY

AND

Qualitative Inorganic Analysis

ADAPTED FOR USE IN THE LABORATORIES OF
COLLEGES AND SCHOOLS

BY

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PREFACE.

THIS treatise was commenced to supply a course of Practical Chemistry to my own classes. I was encouraged to proceed with it by finding that the want of a sufficiently systematic and explanatory Laboratory Text-book was very widely felt.

It has been my aim throughout to give all necessary directions so fully and simply as to reduce to a minimum the assistance required from a teacher. The language employed has, I trust, been rendered simple and intelligible by avoiding the unnecessary use of scientific terms, and by explaining or paraphrasing in ordinary words any such terms when they are introduced for the first time.

The directions for working, and the descriptions of the preparation and use of apparatus, have been given more fully than is usual; my own experience, confirmed by that of other teachers, convinces me that one of the most serious hindrances to the utility of many of the smaller Text-books on Practical Chemistry is the conciseness of the language employed; on this account it is unintelligible to the student unless supplemented by very copious verbal explanation from the teacher.

Whilst making the very desirable amplifications above referred to, the book has been kept within small dimensions, by the omission of all such instruction as does not directly bear on the ordinary requirements of the laboratory student, and by the insertion of the supplementary or merely explanatory portions in smaller type.

I have also thought it best to avoid entering into any lengthy theoretical explanations. The modern teaching of chemistry is in practice very appropriately divided into two departments; namely, theoretical instruction imparted by classes and lectures or by the study of text-books of theoretical chemistry, and practical experience acquired in a chemical laboratory by working according to the directions of a practical text-book. Since by this system the student has time and opportunity afforded him elsewhere for the study of the theoretical and descriptive portions of the science, it is unnecessary and undesirable that his Practical Text-book should tempt him to bestow valuable time in the laboratory upon the consideration of matters which do not directly bear upon his experimental work.

The analytical reactions and methods have been carefully worked through from the text by myself and by the members of my classes: the accuracy and intelligibility of their descriptions have thus, I hope, been secured. Only those reactions and methods which are commonly employed for analytical purposes have been described. In selecting analytical methods, I have felt it desirable that those chosen should be as simple and easy of execution as possible; in some cases, however, methods which are most eligible on these grounds have proved on trial to be so inferior in accuracy and delicacy, that they have been abandoned in favour of others which are recommended by their trustworthiness rather than by their simplicity. In such cases, however, I have also described the more simple methods, since they may be employed in analyses in which minute quantities of a substance have not to be tested for.

I have also felt it advisable to introduce reactions which are useful in general analysis rather than those which claim to be merely interesting and instructive: and I have not hesitated to adopt methods which are not quantitative, if they are easy and accurate for qualitative analysis.

The book is intended to give a course of instruction suited to the general requirements of laboratory students. Those who are using it for any special purpose, or who wish to prepare themselves for any particular examination, should note that the book has been divided into seven sections, the the contents of which are fully stated on pages ix–xv. The first six sections, omitting the fifth, contain a good practical course for senior students; this may however be somewhat modified and amplified to suit junior students, who will possibly also substitute the fifth section for the sixth.

The fifth section has indeed been inserted for the use of those who are preparing for practical examinations in which proficiency in the analysis of simple salts only is required of the candidate. This is the standard fixed for the more elementary examinations conducted by the Universities of Oxford, Cambridge, and London. The sixth section supplies additional details suited for the analytical work of advanced students, and will be found sufficient to qualify a student for the higher University examinations in qualitative analytical chemistry.

The seventh section contains suggestions regarding the fitting and furnishing of the laboratory, together with full lists of all apparatus, reagents, and chemicals required in working through the different sections of the book; there is also added a list of general apparatus, with a description when necessary of its construction and use. In this section there will also be found full and systematic descriptions of the most simple methods for preparing the different solutions required in analysis, with a statement of the strength most appropriate for each. Experience has proved that these are matters which, on the score of economy of time and money alone, merit more attention than is usually bestowed upon them. This section concludes with tables of atomic weights, and of French and English weights and measures.

Symbolic notation has been employed, instead of the full chemical names, throughout the sections on analytical chemistry: in its most concise form this chemical shorthand conduces so much to brevity in writing down results that no other plea is required for its use. The simple plan of labelling each bottle in the laboratory with the chemical formula as well as the name of its contents, will prevent any perplexity to beginners arising from this general employment of chemical formulæ.

The arrangement of all Tables *across* instead of *along* the pages renders the turning of the book unnecessary, a convenience which will be appreciated by all laboratory students. The "Tables of Differences," which contain for each Analytical Group a summary of the differences of behaviour of its members with reagents, will be found suitable for elementary work, and will serve as a key to the Analytical Tables.

It is almost superfluous to mention that free use has been made of the standard works on Chemical Analysis; and a great part of my work has consisted in putting their statements into a systematic and convenient form. My acknowledgments are also due to many experienced teachers who have made valuable suggestions of addition and emendation.

I have been enabled to make improvements in the illustrations of the present edition, by the generous assistance afforded by Mr A. H. Simpson in photographing apparatus for the engravers: and Mr J. B. Coleman has helped me very considerably both by suggestions, and by carefully revising the proofs.

The book is intended to furnish a course of laboratory instruction in practical chemistry which may precede the higher training of the professional chemist and the medical man, and the more special training of the technical chemist and the chemical engineer. It will thus supply a demand which is rapidly increasing, as the value of a sound elementary instruction in practical science is becoming more widely appreciated, both as a means of mental training and as a preparation for a professional career, as well as for many branches of manufacturing industry and enterprise.

F. C.

UNIVERSITY COLLEGE, NOTTINGHAM,
March 1885.

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APPARATUS FOR EACH BENCH-LOCKER.

1	Bunsen's burner with tubing and rose-top (1).
1	Test-tube stand, with twelve holes.
12	Test-tubes.
1	Test-tube brush (11).
2	Boiling-tubes.
2	Round glass plates.
2	Porcelain dishes.
2	Watch glasses.
1	Four-ounce conical flask.
1	Pestle and mortar.
1	Iron tripod stand.
1	Piece of coarse iron wire gauze, or asbestos millboard.
3	Glass funnels, and some cut filter papers.
3	Small beakers.
3	Glass rods.
1	Piece of platinum foil.
2	Pieces of platinum wire.
1	Blowpipe.
1	Pipe-clay triangle.
1	Wash-bottle.
1	Retort stand.
1	Wooden filter stand.
1	Pair of brass crucible tongs.
1	Small horn spatula.
1	Wicker draining basket.

A fuller description of this apparatus will be found on page 318. Most of the apparatus may be seen on the bench in the Frontispiece illustration.

SECTION I.

PREPARATION AND USE OF APPARATUS.

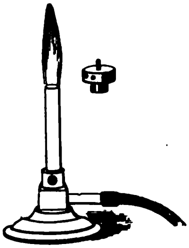
A student who is commencing work in a general laboratory will find on the preceding page a list of the apparatus which should be contained in his private locker; this should be looked through, fitted, and cleansed, as is directed in this section.

In the seventh section will be found particulars of other apparatus, which may be used by many students in common; also lists of the various reagents, and the methods of preparing them.

For convenience in reference, paragraph numbers are printed in the text in thick numbers, and in square brackets at the top of each page.

1. *The Bunsen burner* (fig. 1) is generally employed in the laboratory for heating purposes. It is so constructed that coal gas entering near its base is mixed with a proper proportion of air before being burnt; the air is drawn in through holes at the lower part of the burner, and the mixture of gas and air is burnt at the top of the upright tube. The oxygen of the air, which is thus mixed with the gas, burns the carbon in the body of the flame. Accordingly the luminosity, which depends upon the existence in the flame of unburnt carbon or carbon compounds, is destroyed. The Bunsen flame is therefore mainly useful, because it deposits none of these particles in the form of soot upon a cool object, but also because it is hotter than an ordinary luminous flame. Its high temperature, non-luminosity, and colourless appearance, also render it very valuable for producing flame colorations, as will be seen hereafter. The lamp should be

FIG. 1.



provided with some means for partly or entirely closing the air-holes when requisite; this is usually effected either by turning round a loose perforated ring which is slipped over them, or by rotating the upright tube on its long axis. When a small flame is being used, the supply of air should be partly shut off, else the flame is apt to recede and *burn below.*

When the lamp is to be used, it is connected by means of a piece of tightly-fitting india-rubber tubing, about 5-16ths of an inch in internal diameter, with the tube which supplies gas to the working bench; the tap is then turned on, and in a few seconds the gas is lighted. The flame should be almost perfectly colourless and give scarcely any light.

Occasionally the gas will *burn below*, that is at the bottom instead of at the top of the burner; this usually happens when the flame is small and the quantity of air admitted through the holes is too great; it also occurs when the burner is lighted too soon after turning on the gas tap. A luminous flame may then be seen through the holes in the base of the lamp, burning from the small gas jet inside; and the flame produced at the top of the burner will also be long and somewhat luminous, and will emit a very unpleasant smell, which is due to the incomplete combustion of the gas. In such a case the flame should be at once extinguished by pinching the india-rubber tubing close to the burner, and the gas should be relighted after escaping for a few seconds.

Prove the above statements by trial; study also the effect of partially or entirely closing the air-holes of the burner.

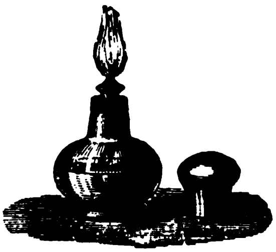
For diffusing heat over a large surface the *rose-top*, which is a small perforated metal cap, is placed upon the top of the burner; it yields a circle of flames, and thus diffuses the heat; the rose-top is put on the burner and removed from it by grasping it with the crucible tongs; it is of course very hot after being used, and should never be handled, or placed upon wood or glass, until it is cool.

For heating large vessels Fletcher's solid flame burner is most convenient and satisfactory; in its smallest size it is also very convenient on the student's working bench.

2. *The spirit lamp* is occasionally employed instead of a gas burner, but for general purposes it should only replace these burners where coal gas cannot be obtained.

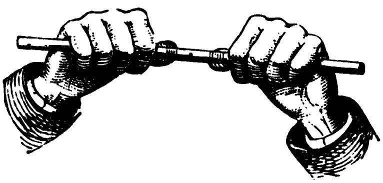
The spirit lamp (fig. 2) consists of a glass vessel containing methylated spirit, into which dips a cotton wick supported by means of a brass or stoneware wick-holder. When not in use the upper end of the wick should be always covered with the glass cap to prevent evaporation of the spirit. If the spirit is tolerably free from resinous matter its flame will be non-luminous, and will deposit no soot upon a cold object.

FIG. 2.



3. *Glass tube or rod is cut* by laying it upon a flat surface, and making a deep scratch with the edge of a three-cornered file at the point to be cut. The glass is then held with both hands, one on either side of the scratch and close to it, and a gentle pressure is exerted upon the glass as if trying to break it across (fig. 3); if the file mark has been made sufficiently deep, the glass will readily break at this point. The sharp edges of a rod or tube should always be at once rounded by holding them in the Bunsen or blowpipe flame until they are partly melted, or by rubbing them with the face of a file.

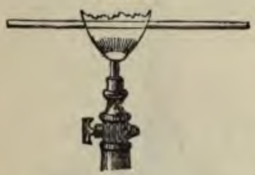
FIG. 3.



Cut off from a length of hard glass tubing which is about one-eighth of an inch in internal diameter, one piece about ten inches, and two pieces about five inches long, and carefully round off their sharp freshly cut edges. Also cut off three pieces of glass rod, seven, six, and three inches in length. Keep these for future use.

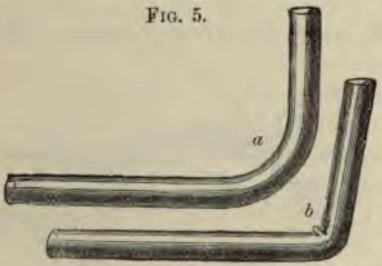
4. *Glass tube is bent* by holding it in the upper edge of a common fish-tail gas flame, so as to heat at least two inches of the glass (fig. 4). The tube is supported by both hands, one on either side of the flame, and is constantly turned slowly round on its axis, so as to heat all sides equally. As soon as the glass is felt to be soft and pliable, it is taken out of the flame and quickly bent to the required angle. The heated part must not be allowed to touch anything until it is cold; the soot is then removed from it by a cloth or a piece of paper.

FIG. 4.



A bend, if properly made, should be a curve and should not alter the bore of the tube (fig. 5 *a*); if a sharp angle is made, the bore will be narrowed (fig. 5 *b*), and the bend, besides being unsightly, will be very liable to break under a small strain. The Bunsen flame must never be employed for bending glass tubing; it is too hot, and melts the glass. *Glass rod*, however, may be bent in the Bunsen or even in the blowpipe flame.

FIG. 5.

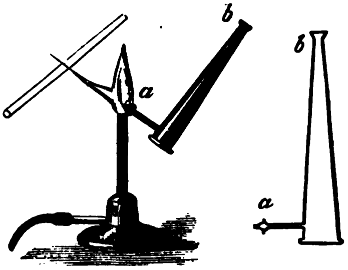


Bend the longest piece of glass tubing (3) at right angles, so that the shorter part is about two inches in length; keep this for future use.

5. *The blowpipe* is frequently used to produce a small but very hot flame from the flame of a gas lamp, spirit lamp, or candle, by blowing through it a fine stream of air from the mouth. It is held in the right hand, with its finely pierced tip *a* (fig. 6) resting on the edge of the burner, and just inside the flame; the mouth-piece (*b*) is then taken between the lips, and after blowing out the cheeks to their

full extent, the air contained in them is forced out through the jet (*a*); this causes a small pointed tongue of flame to issue from the side of the flame. The chief difficulty in learning to use the blowpipe properly is experienced in acquiring the habit of keeping up the blast of air uninterrupted by the breathing. A little patient trial will, however, soon remove this difficulty. It must be borne in mind that the cheeks are to be kept constantly inflated with air, and that the air should be forced through the blowpipe *by the pressure of the cheeks alone and not by the action of the lungs;* breathing is carried on meanwhile through the nose, and the mouth is occasionally replenished with air from the throat just before breathing out through the nose.

Fig. 6.



It is frequently necessary to have both hands free whilst using the blowpipe; this may be secured by resting the jet (*a*) on the top of the burner and supporting the other end (*b*) by the lips alone, as is shown in (fig. 6).

The bright flame obtained by nearly closing the air-holes of the burner is much better suited for blowpipe work than the ordinary non-luminous flame. A burner with an elliptical orifice which gives a flat flame is often substituted for the ordinary Bunsen burner; this flame may also be obtained by slipping a brass tube down the burner.

The student should as soon as possible acquire dexterity in the use of the mouth blowpipe; for long maintaining a high temperature, or for extensive glass-working or glass-blowing, however, a Herapath or Fletcher blowpipe fed with wind from the Fletcher bellows which are blown by the foot, is very convenient and often indispensable.

6. *Small ignition tubes.*—One of the pieces of hard glass

tubing, about five inches long (3), is drawn out at its middle point by heating it strongly in the blowpipe flame, as is shown in (fig. 6). Whilst being heated it is held in both hands as in (fig. 3), and is constantly turned round upon its long axis; when well softened it is gradually drawn out by pulling its ends in opposite directions. The narrowed portion of the tube (fig. 7) is then cut across (3) at its middle point, and by heating the conical part (*a*), the narrow tube may be drawn off, and a small closed tube is obtained (fig. 8).

FIG. 7.

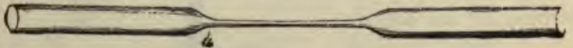
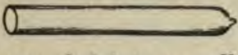


FIG. 8.

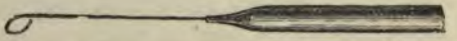


If the closed end is strongly heated in the blowpipe flame, and gently blown into whilst red hot, it may be expanded into a small bulb. Small test-tubes, three inches long by half an inch in diameter, will also serve well for ignition tubes.

7. *Mounted platinum wires.*—Two platinum wires, each about two inches in length, should be fixed in glass handles; they are thus rendered much less likely to be lost, and are far more easily held. The platinum wire used must not persistently colour the flame green.

Draw out the remaining piece of glass tube (3), about five inches in length, at its middle point (figs. 6, 7), and cut it across at the middle of the narrow portion. Each piece of glass thus obtained serves for the handle to a wire. Break off the narrow part of the tube until it extends only about a quarter of an inch from the shoulder *a* (fig. 7); insert the end of the platinum wire into the narrow opening; then hold the end of the tube with the wire in the blowpipe flame until the glass melts and thickens around it, fixing it firmly when cold (fig. 9). The free end of the wire should

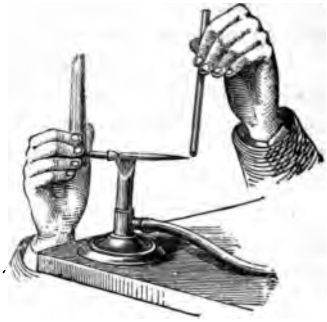
FIG. 9.



then be rolled round a stout wire, so as to shape it into a loop about the eighth of an inch across.

8. *Glass stirring-rods.* — From the ends of the glass rods already made (3) by cutting a length of solid glass rod into pieces about three, six, and seven inches long, any small projections are removed by the file; both ends of each rod are then heated to redness in the tip of the blowpipe flame, the rod being meanwhile constantly turned round on its long axis (fig. 10); the sharp edges are thus removed. The end of the rod must not be allowed to touch anything until it is cool.

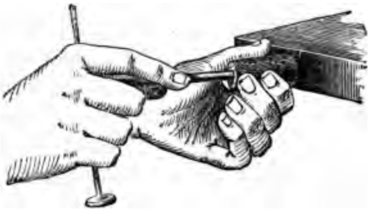
FIG. 10.



Sometimes a very fine glass rod is required; this may be obtained by heating a part of an ordinary rod in the blowpipe flame until it is well softened, and then drawing it out to the requisite fineness (fig. 6).

9. *A cork may be bored* by pushing through it a sharply pointed and slender round file, with a constant twisting movement. The cork-borer, which is a brass tube sharpened at one end, is, however, more commonly used. A borer must be selected of the same size as the glass tube which is to be inserted into the cork; it should be rather less than greater; the cork is then pressed against a wooden surface or grasped firmly in the hand, and the perforation is made by gently pushing the borer through it with a constant movement of rotation back-

FIG. 11.



wards and forwards upon its axis (fig. 11). Caution and practice will enable the student to make a clean straight hole without damaging the surrounding parts of the cork.

In boring a single hole through a cork, the easiest way to make it straight is to bore from the centre of one end halfway towards the other, then reverse the cork and bore a hole to meet this from the centre of the opposite end.

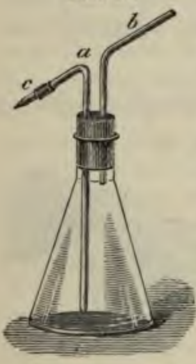
A round file is used for smoothing the interior of holes made by the cork-borer, or for slightly enlarging them when they are too small; whilst doing this, great care must be taken to leave the hole round in shape, and not to enlarge it so much that the glass tubing, when inserted, fits loosely.

In perforating india-rubber stoppers the borer must be sharp, and should be partly filled with water, or moistened with alcohol or glycerine.

The cork-borer is sharpened by rubbing the outer part of the edge obliquely with the face of a fine-toothed file.

10. *The wash-bottle.*—A thin flat-bottomed conical flask, about eighteen ounces in capacity, and not less than an inch in diameter in the neck, is fitted as is shown in (fig. 12).

FIG. 12.



Select a sound cork which is slightly too large to enter the neck of the flask; roll it backwards and forwards under the foot with gentle pressure; when thus softened the cork must fit tightly into the flask. Two pieces of glass tubing, rather longer than would be required for the tubes (a, b), are then bent (4) into the form shown in (fig. 12); their ends are cut off to the right length, and the sharp edges are rounded by holding them in the Bunsen flame, or by rubbing them with the face of the file.

Two parallel holes are then bored in the cork by means of a round file or with a proper sized cork-borer (9); the holes must be somewhat smaller than the glass tubes, and must not run into one another or to the outside of the cork. They are smoothed and slightly enlarged, if necessary, by the round file. Into these holes the tubes (*a*, *b*) are then gently pushed with a twisting motion; they must enter somewhat stiffly, but without requiring much pressure. If the holes have been carelessly made too large, the tubes may often be made to fit by slipping upon them little pieces of narrow india-rubber tubing, or by putting the pieces of india-rubber tubing into the holes in the cork before fitting in the glass tubes.

A vulcanised india-rubber stopper is much more durable than a cork for this and most other chemical purposes; it may be purchased with two holes already made, or may be perforated by a sharp well-wetted cork-borer (9), or by a wetted round file. Both the glass tubes and the inside of the holes should be well wetted before pushing in the tubes, since water serves as a lubricant for glass against india-rubber.

Before completing the fitting of the flask, the cork with the tubes is inserted into its neck; one tube is then closed with the finger, and whilst blowing down the other one, the cork is carefully watched to see that there is no escape of air; a leakage is as a rule easily heard; by wetting the outside of the cork, however, it becomes visible.

If the cork is air-tight, fit upon the upper end of the tube (*a*) a piece of india-rubber tubing about an inch in length; into the other end of this push a short jet (*c*), made by drawing out a piece of glass tubing in the flame (fig. 6); the narrow opening of this jet may be contracted, if necessary, by holding it in the flame for a short time. The neck of the flask should be bound round with twine like the handle of a cricket bat, or tightly covered with a folded strip of flannel; the wash-bottle can then be handled with comfort after water has been boiled in it and the neck has become heated by the steam.

The wash-bottle is now filled about two-thirds with distilled water, and is ready for use; *tap water should never be kept in it.*

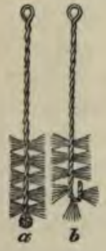
A fine stream of water may be obtained from the jet (*c*) by blowing down the tube (*b*); this stream serves for washing precipitates and for other purposes. If a larger stream is required the flask is inverted, when the water will flow out from the end of the tube (*b*), air entering meanwhile by the tube (*a*); by blowing down (*a*) this stream can be made to issue with greater force and speed.

When hot water is required, the wash-bottle is supported on a tripod stand upon a piece of coarse iron wire gauze, and is heated by the Bunsen flame: or it may be heated by a small Fletcher burner.

11. *Cleaning apparatus.*—It is indispensable to the success of an analyst that all glass and porcelain apparatus should be kept scrupulously clean, and before beginning work the student will do well to clean his set of apparatus as is directed below.

Test-tubes, beakers, and porcelain dishes, are washed in a stream of tap-water, their surface being rubbed meanwhile by the test-tube cleaner (fig. 13 *b*). Occasionally, however, the brush fails to remove strongly adhering stains. A little hot dilute hydrochloric acid will remove most such stains, but it is sometimes necessary to heat a little strong sulphuric or nitric acid in a vessel in order to cleanse it; hot caustic potash or ammonia solution may also often be used with advantage to remove grease. In fact, when removing a substance from a vessel to which it strongly adheres, the student should always consider what the substance is, and then remove it by a liquid in which it is easily soluble. Each article, after having been carefully washed and then repeatedly rinsed out with tap water, should be placed upside down in the small wicker basket to drain, or if required for immediate use it should be rinsed out with a little distilled water; the removal by these means of washing water is necessary, because ordinary tap-water contains dissolved impurities, which might be prejudicial.

FIG. 13.



Apparatus should be washed as soon as possible after it has been used, since the surface is usually more difficult to cleanse after standing. The brush must be cautiously moved as it reaches the bottom of a test-tube, as the glass is very thin and is easily broken by undue pressure.

Test-tube brush.—It will be found that the piece of sponge at the end of the test-tube cleaner (fig. 13 *a*) does not well adapt itself to the bottom of test-tubes and boiling-tubes. A much more efficient end is given to the brush by removing the sponge and bending back the end of the wire stem sharply upon itself at a point just above where the hairs commence (fig. 13 *b*). By slightly curving the part of the stem carrying hairs, the brush will better adapt itself to curved surfaces such as those of porcelain dishes.

Test-tubes containing liquids are placed in the test-tube stand; boiling-tubes may be supported in the wooden filter stand. After being washed they should be placed to drain mouth downwards in the wicker basket, which is to be considered as the receptacle for all *clean* glass and porcelain vessels.

Glass funnels should have their narrow stems cut off to within about half an inch from the shoulder (3); the sharp outer edge must then be removed by rubbing it with the face of a triangular file. The inside of the narrow neck is best cleaned by rubbing it with a common tobacco-pipe cleaner, which is inserted from the shoulder of the funnel so as not to cut the hairs.

Platinum foil and wire are cleansed by boiling them in hydrochloric acid and rinsing off the acid with water; the wire should then be strongly heated for some time in the blowpipe flame, until, on being dipped into pure strong hydrochloric acid, it no longer persistently colours the Bunsen flame. If the tip of the wire cannot be cleansed in this way it should be cut off. Commercial platinum often contains barium, and the wire made from it therefore gives a green colour to the flame; such wire is useless for flame coloration tests and for spectrum analysis.

It is best, however, to keep the platinum foil and wire always in a small beaker containing strong hydrochloric acid diluted with sufficient water to prevent it from fuming; the platinum will then be ready for use at any time after having been simply rinsed with water.

Before putting apparatus away it should be made a rule to wash all glass and porcelain which is not in actual use, and place it in the wicker-basket to drain; the basket is then put away with its contents. *Dirty* apparatus should never be kept in the basket. All iron apparatus should be carefully dried and must be kept in a dry place to prevent it from rusting. *Metal apparatus must never be put into the wicker draining basket.*

12. *Heating porcelain and glass.*—A few general precautions should be observed in heating glass and porcelain vessels to guard against cracking them. The two following rules apply to both glass and porcelain alike:—

A vessel containing a liquid must never be heated by the flame above the level of the liquid inside.

A dry hot vessel must be allowed to cool before pouring in any liquid, or placing it on a cold surface.

Porcelain dishes are generally used for the purpose of boiling or evaporating liquids; they are supported on a pipe-clay triangle placed upon a tripod or retort stand; they may be safely heated by a small naked flame.

Porcelain crucibles are used for containing solid bodies which are to be strongly heated; they are supported in the same way as porcelain dishes; the flame should not be allowed to play steadily upon the bottom of the crucible so as to heat it suddenly, but the burner should be constantly moved slightly from side to side until the porcelain is hot. The crucible should also be allowed to cool slowly on the triangle, as contact with a cold body is very apt to crack it. The crucible and its cover whilst hot are handled by the crucible tongs.

Glass vessels require to be heated more cautiously than those made of porcelain. A large naked flame must never be allowed to play for any length of time on one part of the glass surface. In heating a test-tube or boiling-tube, this *local heating* is prevented by holding the tube obliquely with the lower part in the flame, and either moving it gently up and down, or constantly turning it round on its axis.

Ordinary test-tubes are too narrow for *boiling* liquids in, the liquid being very apt to boil over. Small quantities may be boiled and larger quantities heated short of boiling in a test-tube, but the broader *boiling-tubes* are better suited for this purpose. Test-tubes, if not full of liquid, can be held by the neck whilst being heated, if the tube be held obliquely so that the fingers are not over the flame. All risk of burning the fingers is avoided by bending round the neck of the tube a slip of folded glazed paper or of leather, and pinching the ends together close to the tube.

Glass flasks, such as the wash-bottle, are most safely heated by placing them on a piece of wire gauze on a tripod stand; in some laboratories a sand-bath is available, the flask is then heated by being placed on the surface of hot sand.

SECTION II.

PREPARATION AND PROPERTIES OF CERTAIN GASES AND LIQUIDS.

In this section full directions are given for the preparation of some of the more important gases and for certain instructive experiments which may be made with them. The processes of preparation and manipulation required for these gases are more or less typical; the student will therefore, from the experience gained by performing these experiments, easily prepare and experiment upon other gases from the brief directions given here or in any treatise on chemistry. Two examples of the process of distillation are also appended. The paragraphs in small type may be omitted if time is limited.

The reference numbers enclosed in brackets refer to the paragraphs; these numbers will be found in thick type in the text and in square brackets at the head of each page.

A full list of the apparatus and chemicals required for this section will be found in the seventh section; and the fitting and use of the apparatus has been already explained in paragraphs (1–12).

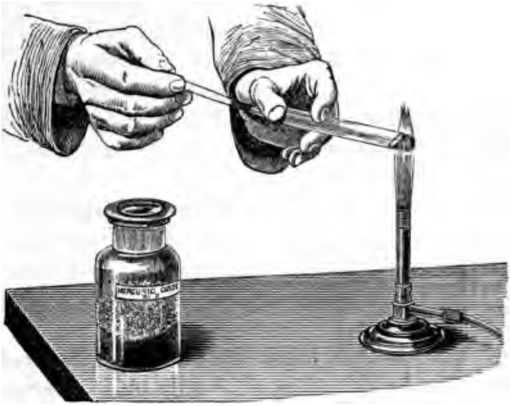
The student must carefully read through *the whole description* of each experiment before beginning to perform it, and after its successful performance a brief description of it should be entered in the note-book. Two students may advantageously, but not necessarily, work together through this section: in the subsequent sections, however, each student should work independently.

OXYGEN GAS.

13. When iron is exposed for some time to moist air its surface becomes covered with rust; many other metals undergo a similar change in moist air, but the alteration produced in their appearance is not usually as noticeable as in the case of iron. The liquid metal mercury does not rust as iron does in moist air, but it becomes slowly covered with red mercury rust when it is strongly heated for some time in a flask open to the air; this mercury rust has received the name of mercuric oxide. The fact that metals become heavier by rusting proves that something is added to them during the process. This is confirmed by the following experiment.

Exp. 1.—Place in a small perfectly dry test-tube sufficient mercuric oxide to cover the bottom; heat the powder in the Bunsen flame (1, 12), loosely stopping the end of the tube with the thumb, as is shown in (fig 14). As soon as small drops of mercury form on the inside of the tube, remove the thumb and quickly insert into the tube the burning end of a thin slip of wood; the flame will be seen to burn more brightly. Heat the powder again for some time in the way just described, and introduce the slip into the mouth of the tube immediately after blowing out the flame and whilst there is a spark at its end; the glowing end will be caused to burst into flame. This behaviour with a burning or glowing slip of wood is one of the most remarkable properties of oxygen gas, and is frequently used as a *test* for its presence. The small

Fig. 14.



globules of mercury may be united by rubbing the inside of the tube; both the globules of mercury, and any red oxide which remains, should be preserved.

The chemical change which has occurred is thus represented by a chemical equation: $HgO = Hg + O$. Since heat can separate mercuric oxide into mercury and oxygen, the process of rusting evidently consists in the metal taking oxygen gas from the air. The increase of weight during rusting, which has been already referred to, is thus accounted for; and if all metal rusts could, like mercuric oxide, be decomposed by heat, oxygen might be obtained from any one of them.

This method of making oxygen is, however, interesting mainly because it was the first means known of preparing the gas; it is never used now-a-days to prepare large quantities of oxygen, since other substances are known which contain a larger proportion of oxygen, and which give it off when they are heated more easily than mercuric oxide does: these substances are also preferable on account of their greater cheapness.

Potassium chlorate is most frequently employed for the preparation of small quantities of oxygen; if heated until no more gas is given off it parts with the whole of its oxygen, as is shown by the following equation: $KClO_3 = KCl + O_3$.

EXP. 2.—Place in a clean dry test-tube a little potassium chlorate and heat it. The white salt after *decrepitating* or crackling, *fuses* or melts, and when further heated appears to boil; the small bubbles which are given off consist of oxygen gas; this may be readily proved by holding in the mouth of the test-tube a burning or glowing splinter of wood.

Potassium chlorate gives off oxygen gas much more readily than mercuric oxide does; but if it is mixed with small quantities of certain other substances, which themselves appear to undergo no change, its oxygen is driven off by heat with extreme facility; of these substances manganese dioxide, or black oxide of manganese, is the one usually chosen.

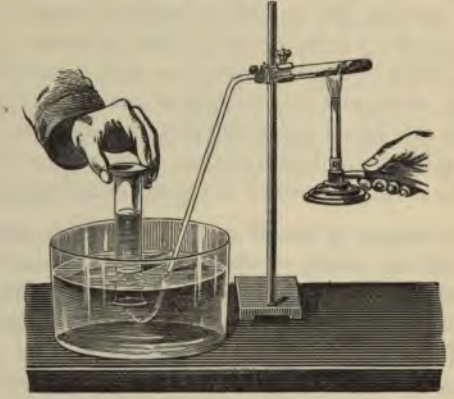
Exp. 3.—Powder finely in a mortar potassium chlorate sufficient to fill a watch glass, mix with it in the mortar about one-fifth as much powdered manganese dioxide, and heat a small quantity of the mixture in a test-tube; the oxygen can be detected by the spark test as soon as the mixture is heated, and a comparatively gentle heat will cause the gas to be rapidly evolved.

In the preceding experiments the oxygen has been detected in the tube in which it was prepared, and has been allowed to pass away freely into the air. When the gas is to be collected in a vessel unmixed with air, the vessel is first filled with water, and the gas is then allowed to displace the water. For this purpose the oxygen is made to pass through a bent glass delivery-tube, which is fitted by means of a cork into the mouth of the test-tube; the end of this tube dips into some water, and the bubbles of gas as they issue are allowed to rise into a vessel full of water which is inverted over the end of the delivery-tube. This process of *collecting* oxygen is fully described in the following experiment.

Exp. 4.—Select a sound cork which is somewhat too large to fit the mouth of the test-tube to be used; soften it by squeezing, or by rolling it on the floor with gentle pressure from the foot; it should now fit tightly into the mouth of the test-tube. Then bend (4) a piece of hard glass tubing, about fourteen inches in length, into the form shown in (fig. 15); so adapting the bends by trial, that when the apparatus is fitted together, the bottom of the test-tube may be at such a height as to be conveniently heated by the flame, the end of the delivery-tube at the same time dipping about an inch under the water contained in a strong stoneware pan. Make a hole through the centre of the cork (9) of such a size that the glass tube fits tightly into it. Then test whether the apparatus is air-tight by fitting the glass tube into the cork, and the cork into the test-tube, and blowing down the open end of the delivery tube; no air must be heard to escape, or must be seen to bubble out on moistening the cork; if air does escape, a fresh cork must be taken. Now transfer the oxygen mixture into the perfectly dry test-tube, either by pouring it off a piece of paper folded into a trough, or by scooping it up from the mortar with the mouth of the test-tube; then fit in the cork and delivery-tube.

Before heating the tube, fill the jar in which the oxygen is to be collected with water, close it with a ground-glass plate, invert its mouth in water three or four inches in depth contained in the stoneware pan or bowl, and carefully remove the glass plate. If this operation has been performed with proper care the jar will be entirely filled with water, and no air bubble will remain.

FIG. 15.



Next proceed to heat the oxygen mixture at the forward part only, since the escape of gas from behind would force the front part of the mixture out of the tube. The tube should either be held in the hand or supported by a clamp with its closed end slightly raised, so that any moisture which may be condensed in the cold end does not trickle back upon the heated portion and crack it. The lamp should be kept slowly moving with the other hand, and a small flame only used, in order to prevent any part of the glass from being suddenly and strongly heated, which would be liable to crack or melt it.

Oxygen gas will soon be evolved, but will not at once appear at the end of the delivery-tube, since it has first to drive out the air which filled the apparatus; as soon as a slip of wood glowing at its end is kindled at the mouth of the delivery-tube, the oxygen has driven out the air and is beginning to escape; the end of the delivery-tube is then at once dipped under water beneath the mouth of the jar, and the stream of bubbles rising in it will rapidly displace the water. A stoneware beehive shelf will be found convenient for supporting the jar: this is shown in (fig. 18). As soon as the vessel is full of gas, close its mouth under water with the glass plate, and remove it for experiment. The jar may also be removed by slipping under its mouth a small dish or saucer, the water taken out in the saucer serving to close the mouth of the jar.

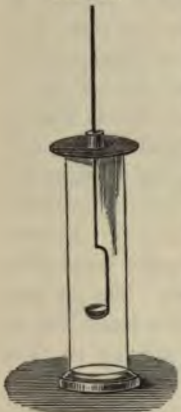
Several jars filled with oxygen will be required for the following experiments, or the same vessel may, if necessary, be refilled with the gas after the completion of each experiment.

If at any time the gas should be given off too rapidly, the flame must be removed until the current of gas slackens; the hinder portions of the mixture should be heated only after the part in front refuses to yield any more gas: when the process is to be stopped, the end of the delivery-tube must be removed from the water before the gas has ceased to bubble out; and the hot part of the test-tube must not be allowed to touch cold or wet objects, which would crack the glass.

Oxygen gas is remarkable for the energy with which it combines with or burns many substances: three examples of this property are given in the following experiments.

Exp. 5.—Select a piece of *wood charcoal* about the size of a hazel nut; place this in a metal deflagrating spoon, then adjust the wire handle in the brass cap, so that when held beside the jar of oxygen with the cap on a level with its mouth, the spoon is about an inch from the bottom of the jar (fig. 16). Now heat the charcoal in the Bunsen or blowpipe flame (5) until a part of its surface glows when held in the air, and quickly place it in the jar of oxygen with the brass plate covering its mouth (fig. 16). The charcoal will burn much more brilliantly and rapidly than in air, and will convert the oxygen into carbon dioxide gas: $C + O_2 = CO_2$. When the combustion ceases, pour into the jar some clear lime water from a small beaker or test-tube, quickly close the jar and shake the liquid inside it; the clear liquid will become milky, indicating the presence of carbon dioxide gas as will be hereafter explained.

FIG. 16.



EXP. 6.—Remove the charcoal from the deflagrating spoon, and replace it by a piece of *sulphur* as large as a pea; heat the spoon in the flame until the sulphur melts and begins to burn with a pale blue almost invisible flame. Then place the spoon in a fresh jar of oxygen; the sulphur will at once burn with a much brighter flame converting the oxygen into sulphur dioxide gas: $S + O_2 = SO_2$. The presence of this gas is proved by its suffocating smell: also by pouring a little water into the jar and shaking it round; sulphurous acid is thus formed, and is recognised by dropping into the water a piece of blue litmus paper, which is immediately reddened; also by pouring in a few drops of red potassium dichromate solution, the colour of which changes to green.

EXP. 7.—Burn away from the deflagrating spoon any remaining sulphur, and put into it when cold a small piece of carefully dried *phosphorus* not larger than half a pea. The phosphorus may be cut with a knife; it must be touched only with the brass tongs or with *wet* fingers, and should be handled as little as possible, since it is liable to catch fire by the heat of the hand; it should always be kept under water, and dried only immediately before being used by pressing it between filter-paper or blotting-paper or with a dry cloth. Set fire to the phosphorus by holding the spoon in the flame and notice how it burns in the air; then place the spoon in a jar of oxygen; the phosphorus will burn most brilliantly, producing a white substance called phosphoric oxide: $P_2 + O_5 = P_2O_5$. When the phosphorus has ceased to burn, pour in a little water and shake it round in the jar; the white substance will dissolve, yielding phosphoric acid: $P_2O_5 + 3H_2O = 2H_3PO_4$. The water may now be proved to contain this acid by dropping into it a piece of blue litmus paper, which will be immediately reddened.

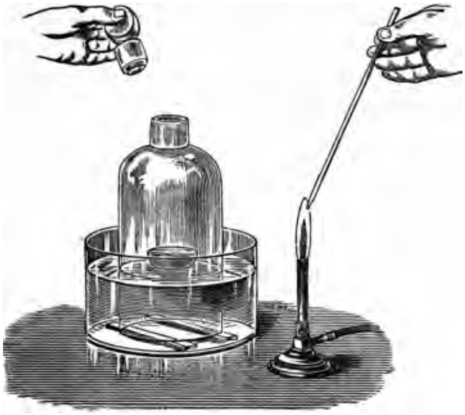
Test for oxygen.—Oxygen is conveniently tested for by introducing into the gas a slip of wood with a spark at the end; the spark is caused to burst into flame. One other gas possesses this property, but it is readily distinguished from oxygen by other means. This test only detects oxygen when it is in a nearly pure condition. Another test for uncombined oxygen in gaseous mixtures is given in experiment 25.

NITROGEN GAS.

14. Atmospheric air has been shown to contain oxygen, but experiment proves that its main constituent is another gas called nitrogen. To obtain this gas phosphorus may be burnt in air enclosed over water; the phosphoric oxide thus formed is allowed to settle down and dissolve in the water; the nitrogen which remains will then be found to amount to four-fifths of the air employed, and to have the property of extinguishing a flame.

EXP. 8.—The air may be most conveniently enclosed in a bell jar, by immersing the mouth of the jar to a depth of several inches in water contained in a stoneware pan, and then inserting the cork or stopper into its neck (fig. 17). A small piece of well-dried phosphorus is placed in a little dry porcelain dish, and the dish is floated on the water in the pan; the open bell jar is then placed as a cover over the floating dish, with its mouth resting on two strips of sheet lead laid upon the bottom of the pan; the phosphorus is kindled by touching it with the end of a long piece of wire, or of a glass rod, which has been heated in the flame; the cork or stopper is then immediately inserted, and the jar is held down by the hand until it stands steadily. When the phosphorus has united with all the oxygen of the enclosed air, it ceases to burn; and as the remaining gas, expanded by the heat, cools, water rises in the bell jar and fills one-fifth of the space previously occupied by the air.

FIG. 17.



Test for nitrogen.—Water should then be poured into the pan until the level inside and outside the bell jar is the same; if the cork or stopper is now removed and a burning taper or strip of wood is introduced, the flame will be at once extinguished by the nitrogen.

OZONE GAS.

15. Oxygen is converted by electricity, and during certain processes of chemical oxidation, into a gas known as ozone. This gas has a peculiar smell, and possesses other properties which distinguish it from oxygen;

since, however, it can be both made from oxygen, and reconverted into oxygen, without any change of weight occurring, ozone is only altered or *allotropic* oxygen.

Exp. 9.—Place a piece of phosphorus in a jar of air, the bottom of which is just covered with a thin layer of water, and close its mouth with a glass plate. Stir a little starch powder with some cold water, and pour this gradually into water which is boiling briskly in a small porcelain dish; cool this starch solution, and mix a few drops of potassium iodide solution with part of it in a little porcelain dish; moisten with this liquid a strip of filter paper, and suspend it in the jar containing the phosphorus. Sooner or later the strip will become blue by the action of the ozone, which has been formed during the spontaneous oxidation of the phosphorus; the faint garlic smell of ozone will also be perceptible in the jar.

HYDROGEN GAS.

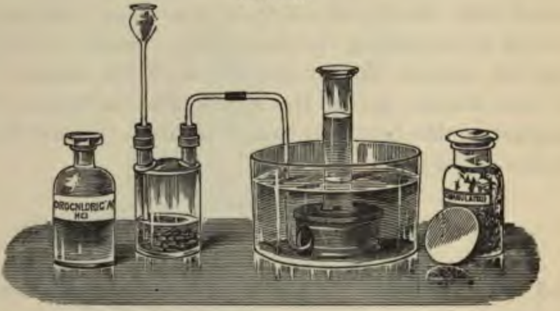
16. Water consists of oxygen combined with another gas called hydrogen; several processes are known for preparing hydrogen from water. This gas is, however, most readily obtained from certain acids by the action of metals upon them.

Exp. 10.—Fit a two-necked Woulffe's bottle with perforated corks, bearing a thistle funnel whose end reaches nearly to the bottom of the bottle and a bent tube terminating just below the cork (fig. 18); then adapt a bent delivery-tube by means of a short piece of tightly-fitting india-rubber tubing. A wide-necked bottle, fitted as is shown in (fig. 21, p. 27), may be used instead of the Woulffe's bottle. Put into the bottle sufficient granulated zinc to cover the bottom; replace the cork; ascertain that the apparatus is air-tight by closing the end of the delivery-tube and blowing down the thistle funnel; then pour water in through the funnel until the zinc and the end of the funnel-tube are covered and add pure strong hydrochloric acid gradually, mixing the acid with the water by shaking the bottle;

hydrogen will be seen rising from the zinc in numerous small bubbles: $Zn + 2HCl = H_2 + ZnCl_2$.

Dip the end of the delivery-tube under water contained in the pan, and allow the gas to bubble out through the water for at least five minutes. This delay in collecting the gas is necessary in order to give the hydrogen time to remove the air which filled the bottle, a mixture of air with certain proportions of hydrogen being explosive if kindled. Before collecting larger quantities, the escaping gas must be proved to be no longer explosive; this is done by inverting a test-tube filled with water over the end of the delivery-tube, closing it with the thumb when it is full of gas, removing it from the water, and holding its open mouth to a flame; if the gas burns with a slight explosion, the tube is again filled with the gas and the experiment is repeated. As soon as the gas burns quietly with a pale flame, a small thick glass cylinder or tube may be filled with hydrogen in the same way as was directed for oxygen; the jar is then removed and the gas kindled.

FIG. 18.



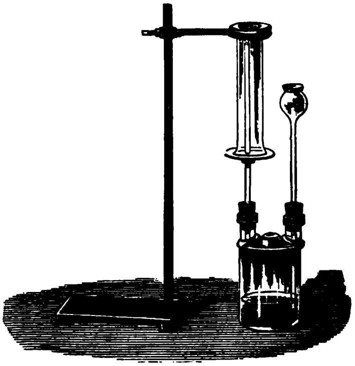
During the further preparation of gas for the following experiments, it is occasionally necessary to pour in a little more strong acid through the funnel-tube when the evolution of hydrogen becomes too slow.

EXP. 11.—Hold another jar of hydrogen for a short time with its mouth open and directed upwards, the gas will entirely escape; the absence of the gas may be shown by holding a lighted taper in the vessel, when no hydrogen flame will be seen. If the cylinder be once more filled with hydrogen and held for a short time with its mouth downwards, the hydrogen will remain in it and will burn with a pale flame when a lighted taper is introduced: note, however, that the gas lights with a slight report; this is due to admixture of air by spontaneous *diffusion*. These experiments prove that hydrogen is much lighter than air, since its tendency to rise prevents it from escaping downwards, whilst it readily escapes upwards.

Since hydrogen is so much lighter than air, it can be made to replace air instead of water in the vessel in which it is to be collected; for this purpose the delivery-tube of the hydrogen apparatus is passed up to the top of an inverted jar, the hydrogen then gradually pushes down the heavier air and fills the jar.

Exp. 12.—A jar may be filled with hydrogen by *displacement* by quickly replacing the bent tube in the cork by a straight one eight or nine inches in length (fig. 19). When a brisk effervescence of gas has been caused by pouring in some strong hydrochloric acid, pass this tube up to the top of an inverted jar, and allow the jar to remain in this position for several minutes. The mouth of the jar may be loosely closed during this process by a piece of perforated metal or cardboard, which rests upon a small piece of india-rubber tubing pushed on the delivery-tube; this will hinder the spontaneous mixture with the air, or *diffusion*, which occurs when the gases are in contact.

Fig. 19.

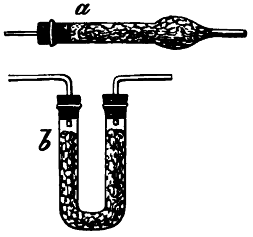


Remove this jar, keeping it inverted, and push up inside it a burning wax taper five or six inches in length; the hydrogen will be lighted and will burn with a pale flame at the mouth of the jar, but the flame of the taper will be seen to be extinguished by the gas: the taper may, however, be rekindled by holding it in the hydrogen flame burning at the mouth of the jar.

Exp. 13.—Cover the bottle and funnel-tube with a cloth to prevent accident in case of an explosion, and light the hydrogen at the end of the delivery-tube used in the last experiment. Hold over the flame a tumbler or beaker, which is perfectly clean, dry, and cool; the inside will become dimmed with moisture, showing that hydrogen gas when burning in the air is uniting with oxygen and producing water: $H_2 + O = H_2O$.

In order to make this result conclusive the hydrogen should be dried before it is burnt, since a gas which has been in contact with water is always *moist*, or contains more or less vapour of water. The gas may be dried or *desiccated* by passing it through some substance which readily absorbs moisture, such as strong sulphuric acid or calcium chloride. A straight tube containing fragments of calcium chloride (fig. 20 *a*), or a U-tube filled with pieces of pumice-stone moistened with strong sulphuric acid (*b*), may be connected with the hydrogen apparatus for the purpose of drying the gas.

Fig. 20.



Exp. 14.—Fill a short thick glass cylinder with water, then invert it in a pan of water; displace one-third of the water in the cylinder with oxygen (13, Exp. 4), and the remainder with hydrogen (16, Exp. 10); let the jar stand with its mouth under water for five or six minutes to allow the gases to mix; then raise the inverted jar and apply a lighted taper: the gases will combine to form water with a loud explosion.

Test for hydrogen.—Hydrogen gas is recognised by burning with a pale flame in air or oxygen, the flame of the dried gas depositing water on any cold object held above it.

CARBON DIOXIDE, OR CARBONIC ANHYDRIDE GAS.

17. This gas was formerly called carbonic acid, a name which is incorrect since all acids contain hydrogen.

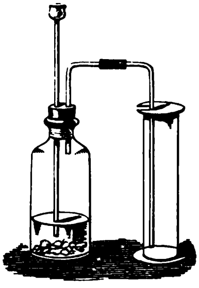
When carbon was burnt in oxygen (13, Exp. 5) carbon dioxide gas remained in the jar: it may be prepared in this way, but is much more easily obtained by pouring hydrochloric acid upon some pieces of marble:

$$CaCO_3 + 2HCl = CO_2 + H_2O + CaCl_2.$$

Chalk or limestone may be substituted for marble, but they are apt to froth and to give off the gas too rapidly.

EXP. 15.—Rinse out the apparatus used for preparing hydrogen and place in it some small pieces of marble; fit into the india-rubber joint a delivery-tube bent at right angles (4), as is shown in (fig. 21); then pour through the funnel-tube sufficient water to cover the marble and the end of the funnel-tube, and add strong *commercial* hydrochloric acid until the gas comes off with brisk effervescence. Place the delivery-tube in a jar with its end nearly touching the bottom, and allow the gas to pass into the jar for several minutes.

FIG. 21.



It is well to cover the mouth of the jar with a small disc of cardboard, which has had a slit or hole cut in it for the delivery-tube, so as to hinder diffusion.

Carbon dioxide, being much heavier than air, will soon fill the jar by displacement; that is to say, the gas constantly supplied to the lower part of the jar will remain there, and, as it accumulates, will gradually lift out the air.

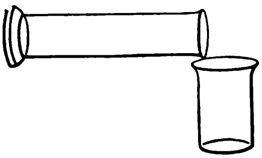
Since this gas has the property of extinguishing a flame, it is easy to ascertain when the jar is full, by holding a lighted taper just inside its mouth: if the flame is extinguished, the carbon dioxide has reached the top.

Carbon dioxide is largely dissolved by water; hence the process of collecting it by displacement is preferred to collection over water.

Exp. 16.—Allow a jar of carbon dioxide to stand uncovered and with its mouth upwards for a few minutes; then place in the jar a burning taper: the carbon dioxide is proved to be still present in the vessel by the immediate extinction of the flame. Now hold the jar for several minutes with its mouth downwards; on testing with a lighted taper, only air will be found in the vessel.

These experiments prove that carbon dioxide is heavier than air, since it remains in a vessel which is open above, and falls out of one which is open below.

Fig. 22.



Exp. 17.—As this gas is so much heavier than air, it can be poured like water into a vessel full of air. A jar full of carbon dioxide is gradually tilted a little beyond the horizontal position (fig. 22), with its mouth over that of a small beaker. After holding the jar in this position for a short time, it may be shown by means of a lighted taper that the gas has left the jar and is present in the beaker.

Exp. 18.—Pour some lime water from a test-tube or small beaker into a jar of carbon dioxide and shake the liquid round in the jar: the lime water will at once become milky. The lime, which was dissolved in the water and was therefore invisible, has been converted by the carbon dioxide into chalk or calcium carbonate; and this, being an insoluble substance, remains mixed as a white powder or *precipitate* with the water: $CaH_2O_2 + CO_2 = CaCO_3 + H_2O$.

Carbon dioxide gas readily dissolves in cold water, forming a liquid which probably contains carbonic acid: $CO_2 + H_2O = H_2CO_3$. This may be proved as follows.

Exp. 19.—Carbon dioxide is made to displace the air from a jar previously half filled with cold water; the mouth of the jar is then tightly closed with the wetted palm of the hand, and the gas and water are shaken together vigorously for a short time; the mouth of the jar will now firmly adhere to the hand, owing to the reduction of the internal pressure caused by the absorption of the gas.

A further proof is afforded by dipping the end of the delivery-tube (fig. 21) to the bottom of a small beaker containing water, so as to cause the gas to bubble through the liquid. After the bubbles have passed for several minutes, a few drops of the water may be poured into a test-tube containing lime water; the lime water will immediately become milky.

On dropping into another part of the carbonic acid solution a little blue litmus solution, or a piece of blue litmus paper, the litmus will become red, indicating the presence of an acid: preserve this liquid.

If the liquid containing carbonic acid be tasted it will be found to possess a taste resembling that of soda water; and in fact soda water is merely water which contains a large quantity of carbonic acid, as may be proved by testing it with lime water and with litmus.

Exp. 20.—Heat some of the water containing carbonic acid in a test-tube until it boils: bubbles of gas will rise in the water as soon as heat is applied, and after the liquid has been boiled briskly for several minutes, it may be proved to be free from carbonic acid by testing it with lime water and with litmus. The reddened litmus in the carbonic acid solution put by from a previous test will also become blue when it is boiled.

These experiments prove that carbon dioxide may be completely expelled from water by boiling the liquid for some time.

Exp. 21.—Dilute some lime water in a small beaker with an equal quantity of distilled water, and allow carbon dioxide gas to bubble through it; a milkiness will be produced at first owing to the formation of calcium carbonate; but if the gas is allowed to pass for several minutes through the liquid, the milkiness will gradually disappear, showing that calcium carbonate is soluble in carbonic acid.

On boiling some of this clear liquid, however, for several minutes, the carbonic acid will be destroyed as has been already shown; the liquid therefore will become milky again, because the chalk can no longer remain dissolved. On pouring out the water part of the calcium carbonate will remain adhering to the inside of the tube; it may be readily removed by rinsing the tube with a little hydrochloric acid.

The above experiment explains the origin of the coating or *incrustation* inside kettles and steam boilers, in which hard chalk water has been boiled. Such water contains chalk dissolved by carbonic acid; this acid is removed when the water is boiled, and a great part of the chalk is deposited upon the inside of the vessel.

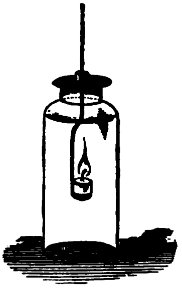
Tests for carbon dioxide.—The presence of carbon dioxide gas may be detected by its power of extinguishing a burning taper, and of turning lime water milky: the latter test distinguishes it from nitrogen. These tests may now be employed to prove that carbon dioxide is evolved from the lungs during the process of respiration, and also that it is produced by an ordinary flame.

Exp. 22.—Invert a jar full of water in a pan of water and blow the breath into it through a glass tube, one end of which is dipped beneath the mouth of the jar. In order to obtain air *from the lungs* a full breath should be taken in, and the greater part having been expelled so as to replace the air contained in the windpipe and mouth by air from the lungs, the remainder of the breath is allowed to bubble up into the jar. Close the jar, remove it from the pan, and introduce into it a lighted taper, the flame will be immediately extinguished.

Fill another jar in the same way and shake up lime water in it; also blow the last part of the breath through a glass tube into lime water contained in a small beaker; the lime water will become milky.

Exp. 23.—Light a small piece of candle or wax taper upon a deflagrating spoon, and place it in a bottle of air the mouth of which is closed by the brass plate (fig. 23). After burning for a short time the flame will be extinguished, and if relighted it will be again extinguished when placed in the jar; this is due to the production of carbon dioxide, and to the reduction of the amount of free oxygen in the air. Now pour in some lime water from a small beaker and shake it round in the bottle, the liquid will become milky. A similar experiment may be performed by inverting the bottle over a small gas flame, and closing the mouth of the bottle with a glass plate as soon as the flame is extinguished. The milkiness produced by shaking lime water in the bottle will again prove the production of carbon dioxide by combustion.

Fig. 23.



Hence carbon dioxide gas is constantly being introduced into the atmosphere by respiration and by combustion, and we should therefore expect to be able to detect its presence in air by the above tests. It is not ordinarily present in sufficient quantity to extinguish a burning taper, but the presence of carbon dioxide in air may be shown by lime water in the following way.

Exp. 24.—Pour some clear lime water into a watch glass or clock glass, and allow it to stand for a few minutes in the air; a film of calcium carbonate will gradually form on the surface, yielding white flakes when the water is stirred.

18.—*Nitric oxide gas* may be prepared in the apparatus already used for making carbon dioxide. The pieces of marble are removed and the apparatus washed out; some copper clippings or turnings are then placed in the bottle, and water is poured in until the end of the funnel-tube is covered; strong commercial nitric acid is then added gradually until a reddish-brown gas appears inside the bottle; this gas is allowed to bubble off for a time through water; it may then be collected over water as was directed for hydrogen gas: $3Cu + 8HNO_3 = 2NO + 3Cu(NO_3)_2 + 4H_2O$.

Nitric oxide is colourless, but it is converted into reddish-brown nitrogen trioxide and nitrogen tetroxide when it is mixed with free oxygen.

Exp. 25.—Prove this by filling a jar with nitric oxide gas over water, and then either passing oxygen up into it, or allowing it to stand with its mouth open in the air; the entrance of the oxygen or air at once produces red fumes.

The appearance of red fumes in the preparation vessel is now explained: the bottle was at first filled with air, the first portions of the gas therefore mingled with free oxygen as soon as they were produced.

AMMONIA GAS.

19. The pungency of common *smelling salts* is due to the ammonia gas which is constantly being given off from solid *carbonate of ammonia.*

The gas is evolved much more rapidly if either *carbonate of ammonia or sal ammoniac* is mixed with lime, and the mixture is then gently heated.

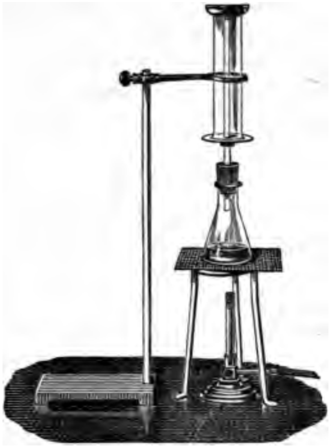
Exp. 26.—Powder a little ammonium chloride or *sal ammoniac* in a mortar, and mix with it about an equal quantity of slaked lime in fine powder: the ammonia gas escaping from the mixture will be readily smelt:

$$2NH_4Cl + CaH_2O_2 = 2NH_3 + CaCl_2 + 2H_2O.$$

Exp. 27.—Pour *Liquor ammoniæ fortissima* into a small flask (fig. 24) until it is about one-quarter filled, and close the neck of the flask with a tightly-fitting perforated cork, into which a straight piece of glass tube eight or nine inches long has been fitted. Heat the flask gently by a small flame upon a piece of wire gauze on a tripod stand: ammonia gas will soon be smelt issuing from end of the tube.

Since it is much lighter than air, ammonia may be collected by displacement in a *perfectly dry* jar; the jar is conveniently supported by passing it through the ring of a retort stand, and letting its mouth rest on a cardboard disc, which is itself supported on a ring of india-rubber tubing as is shown in the figure. To ascertain when the vessel is filled with the gas, it is only necessary to hold at the mouth of the jar a piece of moistened red litmus or yellow turmeric paper; if the ammonia has filled the jar, it will change the colour of the litmus to blue and the turmeric to reddish-brown.

FIG. 24.



Ammonia gas does not burn continuously in cold air, but it burns readily in strongly heated air or in oxygen gas.

EXP. 28.—Show this by holding the end of the delivery-tube, from which a stream of the gas is issuing, in the top of a Bunsen flame; a pale yellowish-green flame of burning ammonia will be seen. If the ammonia is kindled as the end of the tube is passed into a jar of oxygen, the ammonia will also continue to burn.

Ammonia is extremely soluble in water and must therefore be collected either by displacement or over mercury. For this reason the inside of a vessel in which ammonia is to be collected should always be perfectly dry to prevent absorption of the gas.

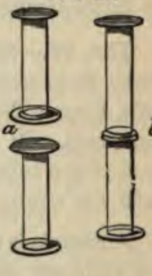
EXP. 29.—Place a jar full of ammonia mouth downwards in a vessel of water, and gently shake the jar so as to agitate the water at its mouth; the liquid will rapidly absorb the gas and rise in the jar until it fills the space formerly occupied by the ammonia: a small space, however, frequently remains filled with the air which was present in the ammonia.

Exp. 30.—Pour a little water rapidly from a small beaker into a jar of ammonia gas, and at once cover the jar with a glass plate or with the hand; then shake the water in the jar; the water, when poured out, will be found to have acquired the pungent smell and the action upon litmus and turmeric papers which characterise the gas.

This liquid is in fact weak *liquor ammoniæ*, a solution which is prepared commercially in large quantities by passing ammonia gas for some time into cold water. It slowly gives off a portion of the gas it contains at ordinary temperatures, and furnishes ammonia gas readily when heated as has been already proved. If ammonia solution is boiled for some time the gas is almost completely expelled from it.

Exp. 31.—Pour into a glass jar a little strong hydrochloric acid, close the mouth with a glass plate and shake the acid about inside the jar, hydrochloric acid gas will thus be liberated; the liquid may then be allowed to run out by slipping aside the glass plate for a moment. Place this jar in an inverted position over another containing ammonia gas and covered with a glass plate (fig. 25 *a*); then withdraw the glass plates, so that the mouths of the jars are in contact (*b*) and the hydrochloric acid and ammonia gases can freely intermingle. Dense opaque white fumes of solid ammonium chloride will immediately be formed: $NH_3 + HCl = NH_4Cl$.

Fig. 25.



These fumes may also be obtained by dipping a glass rod or a strip of filter paper into some strong hydrochloric acid, and holding it in the ammonia gas as it issues from the delivery tube of the apparatus (fig. 24), or in a jar previously filled with the gas.

Tests for ammonia gas.—Ammonia gas may be readily recognised by its pungent smell, by turning moistened red litmus paper blue and moistened turmeric paper reddish-brown, also by giving opaque white fumes when in contact with any surface moistened with *strong* hydrochloric acid.

CARBON MONOXIDE GAS.

20. This gas is produced when carbon dioxide is passed over red-hot charcoal: $CO_2 + C = 2CO$. It is often formed in this way in open grates, and is seen burning with its characteristic blue flame.

A mixture of carbon monoxide with an equal measure of carbon dioxide may be cheaply prepared by heating crystals of oxalic acid with strong sulphuric acid:

$$H_2C_2O_4.2H_2O + H_2SO_4 = CO + CO_2 + H_2SO_4.3H_2O\,.$$

EXP. 32.—Pour upon some oxalic acid crystals in a test-tube about twice as much strong sulphuric acid, and heat the mixture.* In a short time effervescence will be noticed, showing that gas is being evolved; hold in the mouth of the tube a glass rod freshly dipped into lime water; the drop of lime water hanging upon its end will become milky, proving that carbon dioxide is present. Hold a burning taper to the mouth of the test-tube, a blue flame will be produced by the carbon monoxide gas burning in the air.

In order to free the carbon monoxide from carbon dioxide the mixture of the gases is *washed* with solution of caustic soda, or is passed over pieces of solid caustic soda or soda lime; the caustic soda absorbs the carbon dioxide readily, but allows the carbon monoxide to pass on:

$$CO + CO_2 + 2NaHO = CO + Na_2CO_3 + H_2O\,.$$

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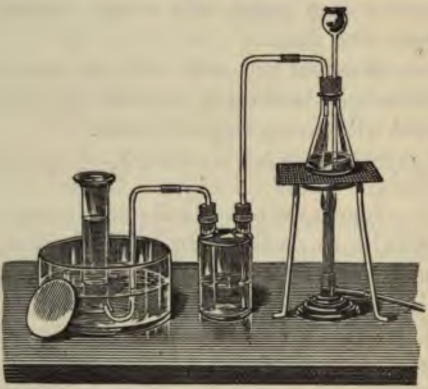
* Strong sulphuric acid is a very corrosive liquid, and great care must be taken not to get any upon the skin, the clothes, or the working bench; should this or any other acid get upon the skin or the bench, it must be at once washed off; if it should be spilt upon the clothes, the part must be immediately wetted with ammonia solution.

If a dilute acid has remained for some time on the clothes it will produce a red stain; this may be removed by ammonia solution, unless it has been caused by nitric acid.

EXP. 33.—Heat a mixture of oxalic acid crystals and strong sulphuric acid in a flask fitted as is shown in (fig. 26), carefully moderating the heat when effervescence commences. Pass the gases which are evolved through a wash-bottle containing caustic soda solution, or through a tube (*a*) or a U-tube (*b*) shown in (fig. 20), which is filled with fragments of slaked lime, caustic soda, or better soda lime, or with fragments of pumice-stone or broken tobacco-pipe stem moistened with strong caustic soda solution: if the carbon dioxide is to be completely removed, the gases must be passed through two or more such tubes or bottles.

FIG. 26.



The carbon monoxide gas may be collected over water; it will be found to give no milkiness when it is shaken with lime water, unless the stream of gas has been so rapid that the caustic soda has not been able to absorb the carbon dioxide completely.

By heating a formate with strong sulphuric acid carbon monoxide alone is given off; it is thus readily obtained free from carbon dioxide: $NaHCO_2 + H_2SO_4 = CO + NaHSO_4.H_2O$.

Carbon monoxide resembles hydrogen in being inflammable and in extinguishing flame; it also explodes when it is mixed with certain proportions of oxygen or air and kindled; hence, before collecting it in a cylinder, the gas should be proved to be free from air by filling a small test-tube and seeing that it burns quietly.

Exp. 34.—Push a burning taper up into a jar filled with carbon monoxide; the gas will burn with a blue flame at the mouth of the jar, but the taper will be extinguished. As soon as the gas has ceased to burn inside the cylinder, pour in a little lime water and shake it about; the liquid will become milky, showing that by the combustion of carbon monoxide in the oxygen of the air carbon dioxide gas is produced: $CO + O = CO_2$.

Test for carbon monoxide.—Carbon monoxide is recognised by burning with a pale blue flame in the air, and producing carbon dioxide which renders lime water milky.

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21. *Chlorine gas* may be made in the apparatus employed for the preparation of carbon monoxide (fig. 26): the washing-bottle may either be dispensed with, or it may be partly filled with water to free the chlorine from hydrochloric acid gas.

Exp. 35.—Place some small pieces of manganese dioxide in the flask, pour in strong hydrochloric acid mixed with about one-third its measure of water, and heat the mixture gently in a draught-cupboard or in the open air. A greenish-yellow gas will be evolved, which may be collected by displacement since it is much heavier than air:

$$MnO_2 + 4HCl = Cl_2 + MnCl_2 + 2H_2O.$$

This gas has a very destructive action on the lungs, and must on no account be inhaled in any quantity.

Tests for chlorine.—Chlorine is usually recognised by its yellowish-green colour, its peculiar smell, and by its property of bleaching moist vegetable colours.

This last property is shown by placing in a jar of the gas a piece of moistened litmus paper, or of *Turkey-red* fabric dyed with madder: the colours of both will be destroyed. A burning wax taper plunged into a jar of chlorine continues to burn with a very smoky flame: a strip of filter paper wetted with hot turpentine catches fire spontaneously in chlorine, and gives rise to dense smoke: a piece of dutch-foil or copper-leaf also burns when dropped into chlorine.

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22. *Hydrogen chloride, or hydrochloric acid gas.*

Exp. 36.—Place in the flask which was used for the preparation of carbon monoxide (fig. 26) several lumps of rock salt or sodium chloride; pour in some strong sulphuric acid and heat gently: hydrochloric acid gas will be evolved: $NaCl + H_2SO_4 = HCl + NaHSO_4$. This gas is heavier than air and should be collected by displacement in the same way as carbon dioxide, since it dissolves easily in water giving liquid hydrochloric acid.

Tests for hydrochloric acid.—This gas is recognised by its pungent smell, by fuming in the air, by turning moistened blue litmus paper red, and by giving opaque white fumes with ammonia gas or strong ammonia solution; it will also render milky a drop of silver nitrate solution, which has been acidified with nitric acid and is then introduced into the gas on the end of a glass rod.

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23. *Sulphurous oxide, or sulphur dioxide,* has been already made by burning sulphur in oxygen; prepare it also as is directed below.

Exp. 37.—Put scraps of copper into the flask (fig. 26), then pour in strong commercial sulphuric acid and heat: sulphurous oxide is evolved when the acid nearly boils: $Cu + 2H_2SO_4 = SO_2 + CuSO_4 + 2H_2O$.

The gas may be cooled and washed by passing it through *a little* water: it is then collected by displacement, since it is much heavier than air and is extremely soluble in water.

Tests.—Show by introducing a burning taper into this gas that it is incombustible and extinguishes flame: note its pungent suffocating smell: pour into a jar of the gas some dilute solution of potassium dichromate and shake it round, also hang paper moistened with the dichromate solution in the jar, the reddish colour is rapidly changed to green: prove also that the gas is freely soluble in water, and that its solution is an acid. Sulphur dioxide is easily detected by these tests.

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24. *Nitrogen monoxide, or nitrous oxide.*

Exp. 38. This gas may be made by heating solid ammonium nitrate: $NH_4NO_3 = N_2O + 2H_2O$. The flask (fig. 26) should be closed by a singly perforated cork bearing a delivery-tube bent twice at right angles. The washing-bottle is replaced by a similar flask closed by a doubly perforated cork; the tubes pass just through this cork, and the flask is weighted by pouring shot into it, so that it may be sunk into a pan of cold water: it serves to condense the steam which escapes with the gas and to cool the gas. Nitrogen monoxide is much heavier than air and may be collected by displacement.

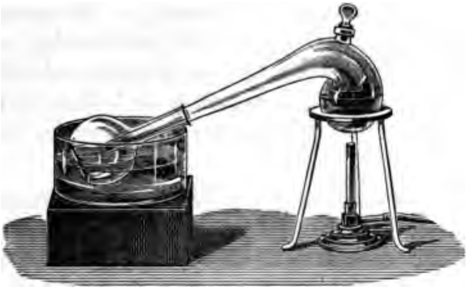
Tests for nitrous oxide.—This gas has a sweet taste; it kindles a spark on a splinter of wood, but differs from oxygen by causing a flame to burn with a greenish halo around it, and by giving no red fumes with nitric oxide.

DISTILLATION.

25. This process serves to separate liquids which boil at a comparatively low temperature either from solids, or from other liquids which are only converted into vapour at a much higher temperature. It consists in boiling the liquid and cooling or *condensing* its vapour into a liquid termed *the distillate;* the non-volatile solid or liquid substances are thus left behind in the vessel in which the liquid is boiled. The purification of common spring water from the solid substances dissolved in it, and the preparation of nitric acid, will serve as examples of this process.

EXP. 39.—*Distillation of water.*—Pour tap water into a clean retort (fig. 27) through a funnel placed in the mouth or in the tubulure, until the bulb of the retort is half full. Support the retort on the ring of a retort stand or upon a tripod stand, with its neck sloping downwards and dipping into a small clean flask, which is partly immersed in cold water contained in a pan or bowl. On carefully boiling the water in the retort, steam will pass into the flask and will be there condensed to *distilled water.* The first portion of the distillate should be shaken round in the flask and thrown away, as it is apt to have been rendered impure by rinsing the neck of the retort and the flask.

FIG. 27.



While this process of distillation is going on, add to some tap water in a test-tube several drops of nitric acid and of silver nitrate solution, and notice that the water becomes milky; this result shows the presence of a *chloride* in the water.

A separate portion in another test-tube will also be found to become milky on the addition of some ammonia and ammonium oxalate solution; the dissolved lime or *calcium* salts produce this turbidity.

The presence of a *sulphate* may be proved by adding to another portion a few drops of hydrochloric acid and of barium chloride solution.

If the distilled water from the flask be examined in the same way it will remain clear, showing that these solid substances have been removed by distillation.

Show also the precipitation of soap by the undistilled water. For this purpose dissolve a small shaving of ordinary soap by warming it in a little distilled water: add a few drops of this soap solution to some tap water which half fills a test-tube; close the mouth of the test-tube with the thumb, and shake the water well: no lather will form, but the water will appear more or less turbid owing to the separation of the soap. Repeat this experiment, substituting an equal quantity of distilled water for the tap water, and adding soap solution to it in the same quantity as before: in this case a lather will form on shaking the water, and the liquid will not become turbid.

Tests for distilled water.—Distilled water should remain perfectly clear when to separate portions of it are added solutions of ammonium oxalate, silver nitrate, barium chloride, and ammonium sulphide: these tests prove the absence of calcium salts, chlorides, sulphates, and of lead and iron salts respectively. Distilled water should leave no residue when it is evaporated to dryness.

Exp. 40.—*Preparation of nitric acid.*—Cleanse the retort from the last experiment by shaking round in the bulb a little dilute hydrochloric acid, and thoroughly rinsing it out with water; let it drain for a few minutes, and then pour in some solid nitre or potassium nitrate; add to this, through a funnel placed in the neck or tubulure, a quantity of strong sulphuric acid about equal in weight to the nitre used; and in case a non-tubulated retort is employed, allow the acid to drain off the neck by supporting it for some time in an upright position. Proceed to distil just as in the preceding experiment; a yellow oily liquid will trickle down the neck of the retort into the flask; this liquid is somewhat impure nitric acid; it will be found to fume strongly in the air: $KNO_3 + H_2SO_4 = HNO_3 + KHSO_4$.

The liquid remaining in the retort should be poured out as soon as it is cool; the retort is then rinsed with water.

Test for nitric acid.—Pour a little of this acid upon some small pieces of copper in a test-tube; reddish-brown fumes will appear in the tube either at once or on gently warming it, an explanation of this result is given under nitric oxide on page 32. This property of giving red fumes when treated with copper is often used as a test for nitric acid.

SECTION III.

ANALYTICAL OPERATIONS.

In all analytical work the *water* used must be *distilled water*, and this only should be kept in the wash-bottle.

Before commencing the analytical reactions, the student should become familiar with the operations which are constantly employed in chemical analysis. The processes will be easily understood by reading through the following descriptions and performing the experiments given in illustration of them.

SOLUTION.

26. Many solid substances, when they are stirred or shaken with water, gradually *dissolve* in that liquid; salt and alum may be mentioned as examples. Other liquids may be employed instead of water, and if they cause solid substances immersed in them to become partially or entirely liquid and to mingle uniformly with the liquid, they are said to *dissolve* the solids. The liquid thus obtained is called a *solution* of the solid, and the liquid which dissolves the solid is termed the *solvent.* Further, a solid which dissolves in a liquid is said to be *soluble* in that liquid; if it does not dissolve it is said to be *insoluble.* Thus water when shaken with sodium chloride or common salt dissolves it, yielding solution of sodium chloride; water is therefore called a solvent for sodium chloride, and this salt is said to be soluble in water.

The process of solution is most rapid when the solid substance in the state of powder is stirred in the liquid, since the largest possible surface of the substance is thus exposed to the solvent; solution is also much hastened by heating the solvent, since this causes a more rapid circulation of the liquid over the solid, and usually increases the solubility of the substance.

Two kinds of solution may be distinguished.

Simple solution occurs when a substance dissolves in a liquid without alteration in composition; the solution, therefore, possesses the taste, colour, and other general properties of the solid; it also yields the solid substance again when the solvent is removed by *evaporation*. Solution of sodium chloride in water is an example of a simple solution.

Chemical solution, on the other hand, is always attended by a chemical change in the substance to be dissolved; the solution therefore contains a substance differing in composition from the undissolved solid, and on removing the solvent by evaporation the original substance is not obtained.

Exp. 41. Place a piece of potassium nitrate in a small clean beaker; partly fill the beaker with water and stir the solid about with a glass rod; the potassium nitrate will *slowly* dissolve in the water; if the liquid be heated by placing the beaker on wire gauze over a small flame, the solution will be much more rapidly effected.

Powder another piece of potassium nitrate by crushing it in a mortar, and then rubbing it round with the pestle; place this powder in a beaker, pour in water, and heat the bottom of the beaker; the potassium nitrate will dissolve much more rapidly than before, showing that solution is accelerated by powdering the solid and by employing heat. Keep these solutions.

Exp. 42.—Powder a little copper sulphate in a mortar, transfer it to a small porcelain dish, half fill the dish with water, and heat it with a small flame upon a pipe-clay triangle placed on a tripod or retort stand. The *blue* copper sulphate dissolves yielding a *blue* solution. Keep this solution.

These experiments are examples of simple solution. The first shows that a colourless solid gives a colourless solution, and the second that a coloured solid gives a coloured solution. This is generally true, and hence the presence or absence of a coloured substance in a solution can often be inferred by merely noting the colour of the liquid.

Moreover if a drop of the potassium nitrate solution be tasted, it will be found to possess the same taste as the solid; chemists occasionally rely upon taste, as well as upon colour, when examining *simple solutions.*

Exp. 43.—Place in a test-tube a small piece of marble or calcium carbonate, pour upon it a little water and heat the tube; the calcium carbonate will be found to be insoluble in water. Add to the water some hydrochloric acid: *effervescence*, or escape of numerous small bubbles of gas, will occur; the piece of calcium carbonate will meanwhile slowly diminish in size, and will at last entirely disappear in the liquid if sufficient acid is added.

Exp. 44.—Place in a test-tube a small piece of copper; on warming this with a little water it remains undissolved; but on adding to the water some nitric acid and heating, the copper slowly dissolves giving off red fumes; it may be entirely dissolved if sufficient nitric acid is employed.

These are two examples of chemical solution; the calcium carbonate is changed by the hydrochloric acid into calcium chloride, and this substance, *not the calcium carbonate*, remains in solution. The copper is changed into copper nitrate, which is then dissolved by the water. It will be noticed that in each of these cases a gas is given off; this is a very usual, but not a universal, effect of chemical solution: the distinguishing fact is that the solid substance has undergone a chemical change in the act of passing into solution.

EVAPORATION.

27. When it is wished to obtain a substance, which is dissolved in a liquid, in the solid condition, the liquid is boiled away as vapour or *evaporated;* the solid substance is then left behind in the vessel.

Exp. 45.—Pour the potassium nitrate solution from (Exp. 41) into a porcelain evaporating basin, and heat it over the Bunsen flame until the water has been nearly boiled away; then make the flame smaller and continue heating until the water has disappeared: the solid potassium nitrate will be left in the dish.

In evaporating a solution, a small flame should always be used towards the end of the process; and to prevent the substance from spirting out of the dish, the flame should be moved about, or the dish may be covered with a round filter paper.

Often only a part of the liquid is evaporated for the purpose of *concentrating* the solution or removing a portion of the solvent. A hot solution, when sufficiently concentrated, will frequently deposit *crystals* of the dissolved substance if it is allowed to cool.

Exp. 46.—Concentrate the copper sulphate solution made in (Exp. 42) and allow the liquid to cool; if sufficient water has been evaporated crystals will form: keep the copper sulphate in the dish.

PRECIPITATION.

28. Two perfectly clear and transparent solutions, on being mixed together, often become more or less turbid or opaque, owing to particles of a solid substance being formed in the liquid: a solid substance thus produced is called a *precipitate.*

Exp. 47.—Pour into a test-tube some barium chloride solution, then add ammonium carbonate solution; a white precipitate of barium carbonate will form: keep this test-tube with the precipitate.

In this instance barium chloride and ammonium carbonate separately dissolve easily in water; but if their solutions are mixed they yield by a chemical change two different substances, ammonium chloride and barium carbonate; the former of these dissolves in water, but the latter is insoluble and therefore remains as a fine powder in the liquid.

A substance is frequently removed from solution by causing it to form an insoluble compound or precipitate; the substances which are added for the purpose are termed *reagents:* they are commonly liquids.

In producing a precipitate, care must be taken that the two solutions are well mixed; mixture may be effected by closing the mouth of the test-tube with the thumb and several times inverting it; or, if the liquid is hot or corrosive and must not be allowed to touch the skin, the liquids may be mixed by warming the bottom of the test-tube in the flame, by stirring the liquid with a glass rod, or by pouring it from one vessel to another.

A precipitate is often caused to appear more rapidly by thoroughly mixing or agitating the liquid, or by warming it.

Precipitates differ much in appearance and properties; and are therefore frequently produced by the addition of suitable reagents in order to show the presence of a substance, or to separate one substance from another.

The appearance of a precipitate is usually described by its colour and its condition: it is *flocculent* if it forms in flock-like masses, *crystalline* if it consists of small particles which are seen to be crystals under a lens or microscope, *gelatinous* if jelly-like. A slight precipitate causes only a *turbidity* in a liquid.

The colour of a precipitate or liquid is often falsified by gaslight; if the colour is to be seen at night, it should be examined with the light produced by the electric arc or by a piece of burning magnesium ribbon.

Occasionally *solid* substances are used to produce precipitates: thus a metal is not unfrequently precipitated from the solution of its salt by the immersion in it of another metal.

Exp. 48.—Dip a clean penknife blade into some of the copper sulphate solution left from (Exp. 46), to which a few drops of sulphuric acid have been added; after a short time the metal copper is precipitated from its solution and covers the iron as a red film.

FILTRATION.

29. This process serves to separate a precipitate from a liquid in which it is suspended.

The liquid containing the precipitate is poured upon porous paper; the liquid itself runs through the pores of the paper, but the solid particles of the precipitate are retained upon the surface of the paper. The paper employed is called *filtering paper* and the liquid which runs through is termed the *filtrate*. A filtrate may frequently be coloured by some substance *dissolved* in it, but it must always be perfectly free from turbidity caused by solid particles *suspended* in it.

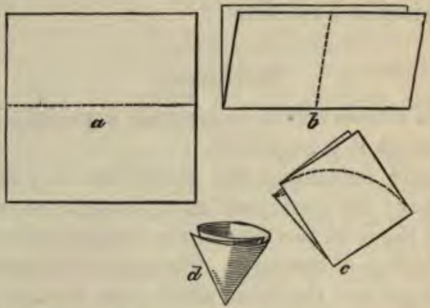
FIG. 28.

Exp. 49.—Measure a glass funnel along its sloping side from shoulder to rim (fig. 28.) Select a circular filter paper, the radius of which is somewhat less than this in length: fold it across, then again at right angles and open it out into the little conical bag (*d*, fig. 29).

If ready cut filters are not at hand, cut a square piece of filter paper whose edge is rather less than double the length of the side of the funnel; fold it over along the dotted line (*a*, fig. 29), then again along the dotted line (*b*); this gives a square (*c*) which at one angle has four free corners; these are removed by cutting with a pair of scissors along the curved dotted line shown in (*c*). The *filter* is now made and only requires to be opened; by separating the curved edges so that they form a circle, three remaining on one side of the circle and one on the other, a little closed pointed paper bag is formed (*d*).

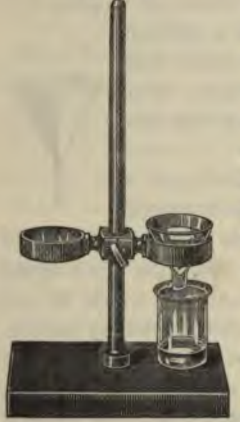
A filter of the same shape may be made from half an ordinary circular paper by folding this into a quadrant, doubling over the radial edges several times, and pressing them down sharply with the finger nail. This dispenses with the unused half of the filter, and renders filtration more rapid.

FIG. 29.



The folded filter is gently pressed *with dry fingers* into the *dry* funnel, and the folding is altered if necessary until the paper fits the glass closely; it is then moistened all over with water from the wash-bottle and is ready for use. This preliminary moistening of the filter paper must not be neglected; since if the liquid and precipitate are poured upon a dry filter, some particles of the precipitate may get between the fibres of the paper, and these, shrinking when they are wetted, retain the solid particles, which choke the pores of the filter.

FIG. 30.



The funnel may be placed with its neck in a test-tube which is supported in its stand; care must, however, be taken that the inside of the upper part of the test-tube is dry, and that there is a space between the neck of the funnel and the inside of the tube, for if the egress of air is prevented the filtration is stopped. The funnel is therefore preferably supported upon the ring of a wooden filter stand (fig. 30); the filtrate being received in a small beaker instead of in a test-tube.

Great care must be taken whilst pouring in the liquid not to let it reach the upper edge of the filter paper.

Exp. 50.—Filter the liquid containing the precipitate of barium carbonate from (Exp. 47). If the above directions have been carried out, and no hole has been made in the paper whilst preparing or fitting in the filter, the filtrate will run through perfectly clear, leaving the barium carbonate on the filter. Keep the funnel with the filter and precipitate.

The filter must always be of such size that when placed in the funnel its edge is below the rim. Sometimes the filtrate runs through turbid; the whole, or the first portions of it, must then be poured once or twice through the same filter; or a double filtration may be performed in one operation by pouring the liquid at once through a double filter.

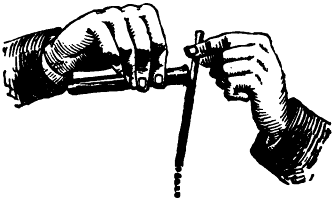
It should be borne in mind that a liquid passes through the filter most quickly when hot; hence, if not inadmissible for other reasons, a solution should always be boiled before being filtered.

Common filter paper contains a little calcium carbonate; this is of no importance in an ordinary analysis, and is only objectionable when an acid solution has to be examined for traces of calcium after filtration. To remove this impurity the filters are moistened in a funnel with dilute hydrochloric acid, and are then well washed with distilled water. Filters may, however, be obtained at small cost which are practically free from soluble matter.

DECANTATION.

30. Precipitates which settle rapidly may frequently be separated without the use of a filter. The vessel is allowed to stand at rest until the precipitate has settled; the liquid is then carefully poured off or *decanted* by gently inclining the vessel: a wetted glass rod, pressed against the edge or lip of the vessel (fig. 31),

Fig. 31.



prevents disturbance of the precipitate. This process, if carefully performed, effects a fair separation of heavy precipitates.

Exp. 51.—Add *dilute* sulphuric acid to some boiling solution of barium chloride; a heavy precipitate of barium sulphate will be formed: this may be readily separated from the liquid by decantation. Keep the precipitate in the test-tube.

WASHING PRECIPITATES.

31. A precipitate which has been separated from the liquid in which it was suspended must usually be *washed* until it is perfectly free from the adhering solution.

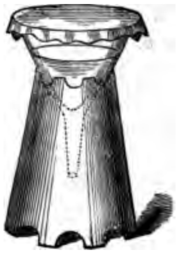
Exp. 52.—*Washing on the filter.*—Support the funnel, which contains the filter and barium carbonate precipitate from (Exp. 50), in a filter stand above a beaker or flask; blow into it a fine stream of hot distilled water from the wash-bottle, so directing the jet as to stir up the precipitate: fill the paper to within a short distance from its edge; let this water run through perfectly, then nearly fill the filter again in the manner just described; repeat this process three or four times, letting the liquid run through completely each time before adding a fresh quantity. The precipitate and filter will usually thus be freed from everything soluble in water, and the water running through will be tasteless. Ascertain whether the washing is complete by collecting the *last few drops* of the washing water in a clean test-tube, and adding to it a drop of silver nitrate solution: no turbidity, indicating the presence of dissolved chloride, must be produced. If turbidity appears, the washing must be continued and the washing water again tested.

Exp. 53.—*Washing by decantation.*—The precipitate of barium sulphate from (Exp. 51) may now be washed by decantation. Hot water is added, and is shaken, stirred, or boiled with the precipitate, which is then allowed to settle; when the water has become clear it is poured off as is shown in (fig. 31). By repeating this washing process several times, with addition of fresh portions of boiling distilled water, the precipitate may be entirely freed from the adhering solution. As the washing water runs from the funnel, test it occasionally with blue litmus paper to ascertain when it ceases to redden the paper and is therefore free from acid.

DRYING PRECIPITATES.

32. A precipitate is dried by placing the funnel in a hollow tin cone or cylinder, called a *filter dryer* (fig. 32); this is supported on a piece of iron wire gauze upon a tripod stand over the flame of a rose-burner turned very low, or upon a heated iron plate or sand bath. The funnel is thus exposed to a current of hot air, which rapidly dries the filter and precipitate. Great care must be taken so to regulate the heat as not to char the filter.

FIG. 32.



A precipitate may be more rapidly dried by draining it in the funnel, then removing the filter and spreading it upon a piece of wire gauze supported on the ring of a retort stand or upon a high tripod stand; a small flame from a rose-burner is then placed beneath the gauze at a sufficient distance to prevent any risk of charring the paper.

A precipitate is partially dried by opening out the filter which contains it upon several dry filter papers, and allowing them to absorb the water: this process may precede those already mentioned.

REMOVING PRECIPITATES FROM THE FILTER.

33. A small quantity of a moist precipitate may be taken from a filter most readily by dipping the end of a glass rod into it; if a watch glass or the interior of a test-tube is then touched with the end of the rod, a portion of the precipitate is deposited for further test or examination.

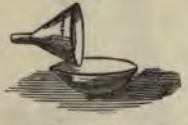
A bulky precipitate may often be taken out of the filter in sufficient quantity by means of a glass or porcelain spatula.

If the precipitate is to be removed from the filter as completely as possible, several methods are available; one or other of those described below must be chosen according to circumstances.

a. A hole may be made in the bottom of the filter by means of a glass rod, which is pushed down through the neck of the funnel: the precipitate is then easily washed down into a vessel beneath by a fine stream of water or other liquid from the wash-bottle.

b. The funnel may be held with its neck horizontal, and with its rim just inside the edge of a porcelain dish (fig. 33); the precipitate is then washed out by directing a fine stream of water from a wash-bottle against the inside of the filter.

FIG. 33.



c. The filter and precipitate are allowed to remain in the funnel for some time in order to allow as much water as possible to drain away; the filter is then carefully taken out of the funnel and further dried if necessary by laying it upon several folds of filter paper; after removing the portions which contain no precipitate, the filter is spread out inside a porcelain dish; the liquid with which it is to be treated is then poured into the dish, and by shaking this round and carefully rubbing the filter with the rounded end of a glass rod, the precipitate is washed off without tearing the paper. The filter paper is then carefully removed by a glass rod.

d. If it is undesirable to add a liquid to the precipitate upon the filter, the filter and precipitate are allowed to drain for a short time, and are then removed from the funnel; the precipitate may be further drained, if necessary, by laying the filter on several dry filter papers. The filter is then spread out upon a flat piece of glass, and the precipitate is carefully scraped off with a glass rod pressed flat upon the paper, or with a small spatula. This method is usually the most imperfect, but is frequently the best for other reasons.

e. If a precipitate is to be dissolved off the filter, the liquid to be used as a solvent is heated and poured upon the precipitate; it will run through the filter into a vessel placed below, taking with it the precipitate in solution. The liquid, after it has run through, should be heated again and once more poured upon the precipitate if the latter is not entirely dissolved; the liquid is reheated and returned to the filter in this way as long as anything remains undissolved; if any portion of the precipitate still remains, it must be removed by the use of a fresh portion of the solvent.

Exp. 54. Remove in this way the precipitate of barium carbonate (Exp. 52) from the filter by means of hot dilute hydrochloric acid.

f. A precipitate may also be removed from the filter by means of the liquid with which it is to be treated or dissolved in the following manner. The funnel is supported over a suitable vessel; the liquid is poured in and quickly stirred up with the precipitate by means of a thin glass rod; the bottom of the filter is then pushed out through the neck of the funnel by the glass rod, and the liquid carrying the precipitate will run through; if some of the precipitate remains on the filter, the liquid is poured again through the filter; by repetition of this process all the precipitate may be removed.

USE OF TEST PAPERS.

34. The two vegetable colouring substances *litmus* and *turmeric* undergo marked changes of colour when they are acted upon by certain substances. These changes serve to indicate whether a liquid or gas is *acid*, *alkaline*, or *neutral*. The analyst is usually provided with slips of paper which have been stained with solutions of these colouring substances: they are known as test papers, and are easily distinguished by their colour. The following experiments will explain their use.

Exp. 55.—*Acid, alkaline, and neutral reaction.*—Dip a clean glass rod successively into dilute hydrochloric acid, ammonium hydrate solution, and barium chloride solution: after each immersion of the rod, draw its end wetted with the solution successively across blue litmus paper, red litmus paper, and yellow turmeric paper: the following results will be obtained.

Hydrochloric acid reddens blue litmus, but does not alter the colour of red litmus or of turmeric: it possesses an *acid reaction.*

Ammonium hydrate solution does not affect the colour of blue litmus, but blues red litmus and browns the turmeric: it shows *alkaline reaction.*

Barium chloride solution does not affect the colour of any one of the test papers: its reaction is *neutral.*

Each of these liquids is typical of a large class of substances as far as its behaviour with these test papers is concerned.

Exp. 56.—*Neutralisation: addition of acid and alkali in excess.*—Pour some dilute hydrochloric acid into a porcelain dish; add solution of ammonium hydrate drop by drop, constantly stirring the liquid with a glass rod and touching the wet end of the rod against a piece of blue litmus paper; by care a point may be reached at which the liquid does not affect the colour of either blue or red litmus paper: the acid is then said to have been *neutralised* by the alkaline liquid. Add another drop of ammonium hydrate and the liquid will acquire an alkaline reaction: the alkaline solution is then said to have been added *in excess.*

It will now be easily understood how an alkaline solution may be neutralised with an acid, and how it may be *acidified* or mixed with *acid in excess.*

The process of neutralising a liquid is most readily completed by the addition at the last of very dilute acid or alkaline solution.

In selecting between red litmus and turmeric test papers *for the* detection of an alkaline reaction, it should be remembered that turmeric is usually more sensitive than red litmus, but that it fades quickly when exposed to daylight.

A very turbid liquid often forms a deposit on test paper which conceals its colour: the colour is, however, usually visible on the back of the paper, or may be easily seen when the deposit is washed off by a stream of distilled water from the wash-bottle.

Test papers must always be kept in a stoppered bottle in order to guard them against change by fumes in the laboratory air: the bottle should be shielded from light as much as possible.

IGNITION.

35. Solid substances are occasionally heated strongly or *ignited* in order to study the effect of heat upon them, since by this means their composition or nature is frequently revealed more or less completely. Ignition also serves to remove volatile from non-volatile substances.

Exp. 57. Heat some zinc oxide in a small ignition tube; its colour will be *lemon yellow* whilst hot, but will change to *white* again on cooling. These changes of colour show the substance to be zinc oxide. In the early stage of the heating drops of water will probably condense in the upper part of the tube, proving the presence of moisture.

Exp. 58. Heat a little mercuric oxide in a small test-tube or ignition tube (6); after a short time minute shining drops of the metal mercury will be seen in the upper part of the tube; if then a small splinter of wood with a spark at its end be held in the tube, it will burst into flame. The behaviour of this red powder on being ignited shows that it contains the elements mercury and oxygen.

Exp. 59. Heat a few particles of charcoal powder on platinum foil by directing the tip of the blowpipe flame against the under surface of the foil; the charcoal will slowly burn away; this behaviour is peculiar to carbon and to a few other combustible substances.

Exp. 60. Heat a piece of ammonium chloride in a small test-tube; the ammonium chloride being volatile will pass off as vapour or *volatilise*, and the vapour cooling in the upper part of the tube will form a coating or *sublimate.*

Exp. 61. Heat in the same way a small piece of potassium chloride; this will melt but will not disappear, since it is only slightly volatile; it will become solid again on cooling.

Hence if a mixture of ammonium chloride and potassium chloride is heated on platinum foil, the potassium chloride alone is left in the residue, since the ammonium chloride is removed as vapour.

Large quantities of substance are usually ignited either in porcelain or in platinum vessels.

It is a necessary condition of *simple ignition* that the substance shall not undergo chemical alteration by contact either with the flame or with the substance of the vessel or support.

FUSION WITH REAGENTS.

36. A substance is often melted or *fused* with certain solid reagents in order to cause chemical changes to occur which yield a characteristic result.

Exp. 62.—Place on a piece of platinum foil a little powdered sodium carbonate and potassium nitrate, and then add a minute quantity of manganese dioxide; hold the foil with crucible tongs in the top of the Bunsen flame, or heat the *under surface* of the foil with the point of the blowpipe flame. The mass will soon melt or fuse, and should be kept in a melted state for a short time and then allowed to cool. A *bluish green* mass will remain upon the foil, the colour of which is due to the sodium manganate which is formed by the fusion; this colour is produced only when manganese is present, and serves therefore as a test for that metal.

In this experiment the solid substances alone react upon one another, the gases of the flame taking no part in the change.

FLAME REACTIONS.

37. The use of the blowpipe has been already explained (5), but hitherto this instrument has been considered only as a means of obtaining a hot compact flame playing in any desired direction.

The gases of either the blowpipe or the Bunsen flame may, however, play the part of reagents in affecting the composition of the substance which is being heated. In order to understand the nature of these changes the structure of the flame must be carefully observed.

In a well formed blowpipe or Bunsen flame two parts may be distinguished: the *inner flame*, which is blue and pointed, and around and beyond this an almost colourless flame known as the *outer flame*. The inner flame is often called the *reducing* or *deoxidising flame*, because it separates or reduces metals from their oxides and from many of their compounds, and in general deoxidises substances; this is due to the action of the hot but unburnt hydrogen and carbon present in this part of the flame. The outer flame is also known as the *oxidising flame*, because it changes metals into their oxides and oxidises many other substances by exposing them to oxygen at a high temperature.

The *inner flame* is best obtained of large size by placing the tip of the blowpipe nozzle only in the very edge of the flame: whilst the *outer flame* is better developed when the nozzle is pushed much further into the flame. The reducing flame is also furnished most readily when the gas flame is made somewhat luminous by partially closing the air-holes of the Bunsen burner: the oxidising flame is most perfectly formed when the Bunsen flame contains much more air.

In the following processes the results obtained will usually vary according to the part of the flame in which the substance is heated.

38. *Fusion with borax: the borax bead.*—Certain metals and their compounds when fused with borax are converted into coloured borates: the colour of each of these borates is characteristic either in itself or in the changes which it undergoes in the outer or inner blowpipe flame.

EXP. 63.—Place a little powdered borax on a watch glass; moisten the loop of a piece of mounted platinum wire (7) and dip it into the borax; then hold the loop with the adhering powder in the outer blowpipe flame until the borax melts. If the bead thus formed does not fill the loop, dip the melted bead again into the borax; more powder will adhere and may be melted in the flame. In this way powder is to be constantly added until a drop or bead is obtained which completely fills the loop. This bead must be perfectly colourless and transparent when it is cold.

If the melted bead easily drops from the wire, either the loop or the bead is too large. A loop which is somewhat less than an eighth of an inch across answers well; it is made by rolling the tip of the wire round a piece of rod. If the bead is too large, remove a portion by giving the wire a sudden jerk whilst the borax is in a fused condition.

If the bead shows any colour, it is jerked off whilst fused and another bead is made from fresh borax; this process is repeated if necessary until a bead is obtained, which, after having been heated in the outer blowpipe flame, is colourless both hot and cold.

Moisten the clear cold borax bead and dip it into some finely powdered manganese dioxide; *a few minute particles* only are to be thus attached to the bead. Then fuse this substance into the bead by heating it for a short time in the tip of the *outer* blowpipe flame, and at once examine the colour of the bead by looking through it at a piece of white paper or at a window light or bright flame; it will be *purple or amethyst red* whilst hot, and its colour will not alter on cooling. Now heat the bead again for some time in the tip of the *inner* blowpipe flame; its colour will vanish, but will reappear when the bead is heated at the tip of the outer blowpipe flame. These changes of colour in the borax bead characterise the metal manganese.

A beginner often obtains at first a bead which is opaque, owing to the use of too much manganese oxide: if this should happen, the bead may be fused and a portion of it shaken off and replaced by fresh borax.

The bead is detached while it is liquid by giving the wire a sudden jerk; it may also be removed when cold by crushing it on a hard flat surface by a sharp blow with the pestle.

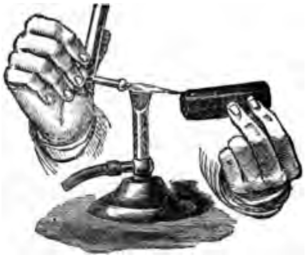
Two mounted platinum wires should be kept, their loops when not in use being always immersed in dilute hydrochloric acid; they will then only require to be washed with water before being used.

39. *Ignition on charcoal in the blowpipe flame.*—Wood charcoal, on account of its cheapness, its infusibility, and its non-conducting power, is a very useful support for substances which are to be strongly heated in the blowpipe flame.

It is often, however, selected on account of the reducing or deoxidising power which it can exert at a high temperature, which renders it entirely unsuitable for oxidising processes such as that described in (Exp. 62).

Ignition on charcoal therefore serves chiefly to detect the presence of the heavier metals: these are smelted out by the reducing action of the inner blowpipe flame aided by that of the red-hot charcoal. The assistance of other solid reagents is, however, often necessary or at least advantageous. The vapour of some metals burns as it leaves the charcoal, and the metallic oxide is deposited as an *incrustation.* The white ash left by combustion of the charcoal must not be mistaken for such an incrustation.

Fig. 34.



Exp. 64.—Choose a piece of wood charcoal which is free from large cracks, and which does not crackle or spit when heated in the blowpipe flame; scoop out a small hollow near one end of it with a knife or with a charcoal borer; place

wire must be boiled with hydrochloric acid in a test-tube and then rinsed well with water; or it may be wetted with strong hydrochloric acid, and heated strongly in the blowpipe flame until it no longer colours the flame. If the loop cannot be cleansed in this way, the end of the wire must be cut off and a fresh loop formed.

Now dip the wire loop into a little sodium chloride solution, made by dissolving some sodium chloride in a few drops of water on a watch glass, and see on removing the loop that it is filled by a drop of the solution; then hold the loop at the tip of the inner blowpipe flame, the outer flame will be coloured *bright yellow*.

Repeat the experiment, placing the loop containing the solution in the outer part of the Bunsen flame about halfway between its base and top; the same yellow tint will be seen above the loop.

This yellow coloration is given to the flame only by sodium and its volatile compounds: on looking at it through the indigo prism, no yellow coloration will be visible.

Remove all sodium chloride from the platinum wire loop by heating it strongly in the tip of the blowpipe flame until it ceases to impart any colour to the flame. Then dip the wire loop into some solution of *pure* potassium nitrate: this will impart to the flame a *pale lilac* colour, which is characteristic of potassium and its compounds. On looking at this coloration through the indigo prism, it will appear *crimson*.

Exp. 67.—Now dip the wire loop into a mixture of a few drops of the sodium chloride and potassium nitrate solutions made on a watch glass; when the loop is held in the flame the sodium coloration only will be seen; but if this coloration is then viewed through the indigo prism, the crimson potassium coloration will be seen distinctly. By means of the indigo prism potassium is therefore discoverable even in the presence of sodium, which conceals its coloration to the naked eye.

THE SPECTROSCOPE.

42. This instrument furnishes by far the most certain means of detecting substances by their flame colorations, more especially when the colorations due to several substances are mingled in the flame. The coloured light is separated into its constituents by passing through one or more prisms in the spectroscope; the coloured lines thus seen are termed the *spectrum* of the substance, and by their colour and relative position they serve to identify the substance with certainty. This is equally true whether the flame coloration is produced by one substance only or by several, since the constituent colours are in any case seen without interference one with another.

Two forms of this instrument are figured here. The direct vision pocket spectroscope, which is shown ready for use in (fig. 37), is recommended by its portability; whilst the Bunsen or table spectroscope, shown in (fig. 38), is to be preferred for general use as a stationary piece of apparatus in the laboratory. The light enters each of these instruments through a slit, the width of which can be adjusted to suit strong or feeble colorations: the rays of light after entering are rendered less divergent by traversing a lens, and then pass through one or more prisms; they are finally focussed by means of a sliding eye-piece, so as to give a sharp image of the slit.

Exp. 68.—The student should now proceed to work with either or both of these instruments.

The direct vision spectroscope is arranged for use by supporting it in a clamp steadily in front of the flame (fig. 37); the slit is then slightly opened and the eye-piece so adjusted that the yellow line, due to the sodium compounds present in the atmospheric dust, is sharply focussed. Or the instrument may be held in the hand and directed on the flame, in this case it is most readily focussed on the dark Fraunhofer lines, which are seen on looking through a window at ordinary daylight.

The Bunsen or table spectroscope (fig. 38) has usually three brass tubes: one of these carries the adjustable slit, another the eye-piece, and the third a transparent photographic scale. The instrument is brought into adjustment by placing a Bunsen burner with closed air-holes in front of the slit; on looking through the eye-piece at this luminous flame a *continuous spectrum* of unbroken colours will be seen: the upper and lower edges of this spectrum are sharply focussed by moving the eye-piece: and the two tubes are arranged at such an angle as to give the longest

Fig. 37.

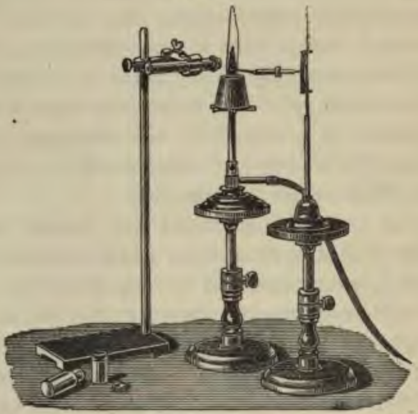
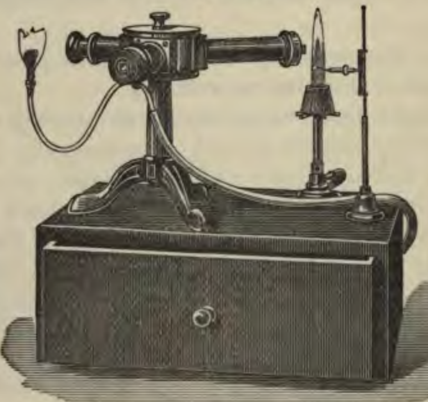


Fig. 38.



complete spectrum possible. The tubes must now either be permanently clamped, or so marked that they can at any time be brought readily into the same position with certainty. Air is then admitted into the Bunsen flame by opening the holes of the burner, and the yellow sodium line, which is always visible, is sharply focussed. The bright flame is lighted opposite the end of the scale-tube, the scale is focussed, and the scale-tube is shifted until the yellow sodium line stands at the fiftieth graduation. This line will of course be seen in all the following experiments.

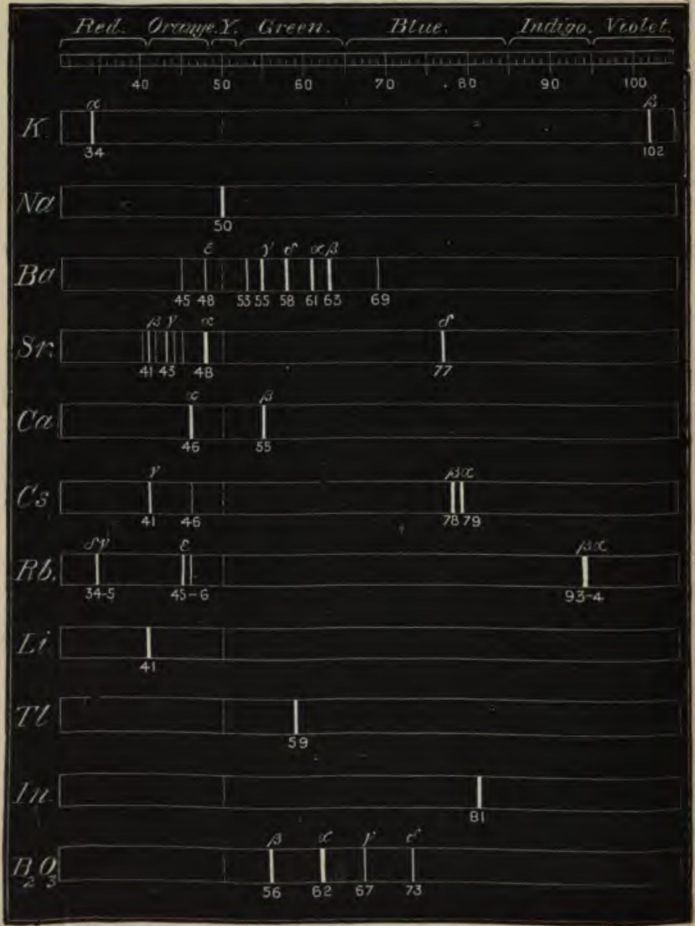
Having thus brought the instrument into adjustment, the student should proceed to introduce into the front of the Bunsen flame and just below the slit, a platinum wire loop moistened successively with solution of sodium chloride, potassium chloride, barium chloride, strontium nitrate, and calcium chloride. The wire may be conveniently supported in the flame by slipping its glass handle upon a horizontal wire arm, whose height is adjustable on a stand such as is represented in (figs. 37, 38). Coloured lines will be seen which are always the same in colour and position for the same coloration, but vary in these respects with the colorations of different substances. This *line spectrum* is therefore characteristic of each vapour which colours the flame.

The pocket spectroscope is not usually furnished with a scale; if it is not, the relative position of the lines of any particular spectrum can only be jotted down or remembered with approximate accuracy; since the yellow sodium line is never absent, it will furnish a useful landmark in the spectrum.

The scale of the larger spectroscope enables the position of the lines to be marked down, and they are thus easily identified again if the instrument is brought invariably to the same adjustment. The lines, however, will vary in position in different instruments, owing not only to varying adjustment, but to differences in the material and angle of the prism. Hence, when mapping down the lines in a spectrum chart for future use, the instrument must always be set in precisely the same way if the chart is to be of any use: and further, a separate chart is necessarily made for each spectroscope.

The accompanying spectrum chart (fig. 39.) will show the student a convenient method of mapping the lines, but it is only intended for use with the spectroscope for which

Fig. 39.



it was made. In this chart the varying intensity of the *lines* is indicated by their varying thickness, and their colour by the name of the colour over the region in which the lines occur; the position of the sodium line is marked in each spectrum. The student should carefully make such a chart, marking in the lines with pen or pencil on a white ground, and placing against each line the number of the scale graduation at which it stands; the relative intensity of the lines should also be indicated by the Greek letters applied in the order of decreasing intensity.

It will be found convenient to register this chart in the note-book, or to suspend it near the instrument.

A spectrum may be identified, even when the instrument has not been carefully adjusted, by throwing in a known coloration from another flame through a part of the slit by means of a little reflecting glass prism, and observing whether the unknown lines exactly correspond with those of the known coloration.

Exp. 69. After the student has carefully examined and mapped the spectra of several of the metals mentioned in the spectrum chart, he should examine some mixed spectra obtained from solutions containing the salts of two or more of these metals; the ease with which metals can be found by means of the spectroscope in the presence of one another will thus become evident.

The delicacy of this method of detecting certain metals is extreme. Thus it is only in the stillest air, which is almost absolutely free from dust particles, that the sodium spectrum is not obtained from a Bunsen flame. By using extremely dilute solutions of metallic salts, this delicacy may be proved in the case of other metals. Bunsen states that one three-thousand millionth of a gram of sodium can be detected by the spectroscope.

It is by no means a matter of indifference which compound of a metal is selected for obtaining its flame coloration or spectrum. The most volatile salts, such as the chlorides, nitrates, and chlorates, give the most vivid but least durable result; whilst the coloration is increasingly feeble with the sulphate, carbonate and phosphate; and is not usually obtainable from the silicate. In most cases a coloration becomes more intense if the finely powdered substance is moistened with strong hydrochloric acid; but in the case of a silicate it is usually necessary to moisten the powder with hydrofluoric acid, or to mix it with pure ammonium chloride and calcium carbonate, before introducing it into the flame.

The width of the slit is regulated according to the intensity of the lines to be looked for; a bright line can be observed with a narrow slit, and its position is then noted with great precision. The slit must, however, be opened much more widely to see a faint line, such as Kβ. Great breadth of the lines is objectionable, since it prevents their position on the scale being accurately noted, and often causes the edges of neighbouring lines to overlap.

The background of the flame should be absolutely black, to prevent admission of stray light; a piece of black velvet hung behind the flame gives a good background.

Careful observation of the flame through the spectroscope is necessary *from the moment* that the substance is introduced, until the wire loop has been heated to whiteness for some time, since some very volatile substances give only a momentary flash of colour in the flame, whilst others, which are far less volatile, colour the flame only at the highest temperature obtainable.

The spectroscope is of special use in analysis:—

1. For readily detecting metals when their flame colorations are concealed by that of sodium; and generally for detecting metals when their colorations are mixed in the flame.

2. For distinguishing similar colorations from one another: thus the red coloration of strontium is readily distinguished from that of calcium by the strongest red line in their spectra being at different distances from the sodium line, and further by the characteristic blue line of strontium and the green line of calcium.

3. For detecting minute traces of many metals which

could scarcely be discovered by other means. The metals lithium, indium, thallium, rubidium and cæsium were discovered by their spectra; and as they usually occur in minute proportion only, they are most conveniently detected by the spectroscope.

In the more powerful spectroscopes several prisms are used, and the lines of the spectra become more widely separated: their position may then be noted with great precision by moving the observing tube until each line in succession is brought into the middle point of the cross wires in the eye-piece; the position of the eye-piece is then read off on the graduated edge of the circular table on which the prisms stand, and registers the relative position of the line. Instruments of great power and precision are however not requisite for ordinary analytical work.

SECTION IV.

ANALYTICAL REACTIONS.

The student will find many paragraphs in this section marked with an asterisk (*): these may be omitted if the analysis of simple salts only is to be learnt. They will, however, require the careful attention of the general analyst.

Reactions printed in small type are of minor importance only.

INTRODUCTORY REMARKS.

The study of chemistry has revealed to us the fact that every substance consists either of one kind of matter only, or of two or more different kinds. Those substances which contain only one kind of matter are called *chemical elements;* their number at present amounts to a little over sixty. A list of them is given at the end of the book (562). These elements do not usually occur singly; two or more of them are commonly found together either in a state of mere *mixture*, or united in a much more intimate manner in a *chemical compound.*

It is the object of analytical chemistry to discover what element, elements, or sometimes what groups of elements, any substance of unknown composition contains; also to ascertain whether any particular element or group of elements is present in a given substance.

In order to become a thorough analyst it is necessary to be acquainted with the properties of each element and of the compounds it forms with other elements; and efficiency in analysis therefore depends in a large measure upon the knowledge of what is usually termed *theoretical*, but more appropriately *descriptive chemistry.* For ordinary analytical purposes, however, certain marked properties or changes each element or group of elements are selected, and by these properties, called *tests* or *reactions*, its presence is detected.

The tests or reactions which are most frequently selected depend upon the behaviour of a substance when heated under various conditions, and upon its behaviour when mixed with certain liquid or solid substances.

Such substances, added for analytical purposes, are called *reagents*. Some reagents serve for detecting one element or compound only, and are therefore called *special reagents*: others separate or detect a group of elements or compounds, and are called *group reagents* or *general reagents*.

Analytical groups.—The metals are separated by their behaviour with certain general reagents into five principal groups, two of these being further subdivided into two subgroups. Each group receives a distinctive name from its place in the system, from some conspicuous or important member of the group, or from the general reagent used to precipitate the group. Thus we speak indifferently of the Fourth Group, the Barium Group, or the Ammonium Carbonate Group. At the beginning of the fifth section the analytical groups are tabulated, with their distinctive numbers, names, and group reagents placed at the head of each column: the names and symbols of the metals contained in the group are arranged vertically beneath. It is perhaps well to avoid referring to a group by its number, as different analysts number the groups differently, and the name thus given is therefore apt not to denote the group with certainty.

Analytical classification.—The chemical elements are frequently divided into two classes, *metals* and *non-metals*. These classes, with the following slight modifications, form convenient divisions for analytical purposes. In the class of metals must be included the hypothetical metallic radicle ammonium, and the element hydrogen. The class of non-metals, either singly or in groups formed by their combination *inter se* or less frequently with a metal, unite with hydrogen and with metals forming *salts*; the hydrogen salts of these radicles are usually called *acids.* Thus Cl, SO_4, PO_4, MnO_4, form the hydrogen salts or acids, HCl, H_2SO_4, H_3PO_4, $HMnO_4$; and the metallic salts, KCl, $MgSO_4$, Na_3PO_4, $KMnO_4$. *Metal hydrogen salts* or *acid salts,* such as $NaHSO_4$, Na_2HPO_4, are also known. The general name *acid-radicle* is conveniently applied to Cl, SO_4, PO_4, MnO_4; some of these are elements, but most of them are unisolated *compound radicles,* and their common property is that of forming acids and salts by combining with hydrogen and with metals. For analytical purposes then, substances may be separated into the groups of *Metals* and *Acid-radicles.*

THE ANALYTICAL REACTIONS.

43. In order to become fully acquainted with the behaviour of different substances with *reagents,* and thus to be able to detect them by their *reactions,* each of these reactions should be tried with the substance itself or with one of its compounds; the appearances presented are then carefully observed, so that the substance would be easily recognised in a body of unknown composition by means of these tests.

The addition of a reagent to a substance causes some important and visible change to occur; this may be simply a change of colour, but it very usually consists in the formation of a precipitate; the colour, appearance, or behaviour of this precipitate with other reagents being characteristic of a particular element or compound radicle.

As soon as a reaction has been satisfactorily performed, it should be briefly described in the note-book in a neat and concise manner; and the nature of the chemical change which has occurred should then be explained by means of a chemical equation. This will involve the correct use of chemical symbols and formulæ, and a right conception of the meaning of an equation. For full information on these matters a treatise on theoretical chemistry should be consulted; the few hints which follow will, however, serve to guard and guide a student who has not had much experience in such matters.

Chemical notation.—Each element is represented by a *symbol* consisting of one or two letters; thus S stands for sulphur, Cl for chlorine. The symbol also represents *one atom* of each element; when written one after another symbols give the *formula* of a compound substance, showing what elements, and how many atoms of each of these elements, the compound molecule contains: thus KCl stands for one molecule of potassium chloride, a compound of one atom of potassium and one atom of chlorine; $PtCl_4$ stands for one molecule of platinic chloride, which consists of one atom of platinum combined with four atoms of chlorine, the small figure placed *below a symbol* to the right denoting how many atoms of that element are present. A number placed *before a formula*, however, multiplies each symbol in that formula; thus $2PtCl_4$ signifies two molecules of platinic chloride, containing altogether two atoms of platinum and eight atoms of chlorine. If two or more symbols are included in brackets, any number placed outside the brackets, whether it be *before* or *after and below* them, multiplies each symbol contained in the brackets; thus both $Sr(NO_3)_2$ and $Sr2(NO_3)$ stand for SrN_2O_6, and this may also be written $Sr2NO_3$.

In the seventh section of this book will be found a list of the elements, with their symbols and atomic weights; also lists of the names and corresponding formulæ of most of the principal compound substances. Symbols and formulæ are always used in the text instead of names; the student can readily find the names corresponding to them either by reference to the lists already mentioned, or by looking at the labels on the reagent bottles.

Rules for drawing out a chemical equation.—Write down the formulæ of the substances which are mixed together, with the sign of addition (+) between them; then write the sign of equality (=), followed by the formula of the precipitate or product formed. In most analytical reactions a mutual exchange of certain elements or groups of elements, or a *double decomposition*, occurs between the two compounds;

a little consideration will therefore usually show how many molecules of the mixed substances are required to yield the formula for the precipitate or principal product; it will also be seen whether any other substance is formed at the same time; and if it is formed, its formula may be easily deduced.

The number of atoms of any one element on both sides of a correct equation must always be equal.

Applying the above rules to the first test for potassium (47), we get:—

$$KCl + PtCl_4 = \overline{K_2PtCl_6}.$$

This statement is obtained by placing together the formulæ of the substances mixed and of the precipitate formed; it is evidently not correct, since there are two atoms of K on the right hand side and only one on the left, also six atoms of Cl on the right and only five on the left; but this inequality is at once removed if we place 2 before the KCl, thus:—

$$2KCl + PtCl_4 = \overline{K_2PtCl_6}.$$

Evidently if the above equation is correct the precipitate is here the only substance formed, since the number of atoms in the substances on the left hand side is exactly equal to that in the substance on the right.

Take now the first test for Ba (69): our tentative statement is:—

$$BaCl_2 + Am_2CO_3 = \overline{BaCO_3} +$$

But here the left hand side contains Am_2 and Cl_2 more than the right; the equality is easily restored thus:—

$$BaCl_2 + Am_2CO_3 = \overline{BaCO_3} + 2AmCl$$

which states that on mixing together barium chloride ($BaCl_2$) and ammonium carbonate (Am_2CO_3), barium carbonate ($BaCO_3$) and ammonium chloride ($AmCl$) are obtained.

In writing out these equations a systematic method should be observed. Write down on the left hand side of the equation first the formula of the substance upon which the reaction is being tried, then that of the reagent added; and on the right hand side write first the formula of the precipitate or principal product with a line drawn over it by way of distinction, then that of any substance or substances which may be formed at the same time.

In the reactions which follow, the formulæ of the substance used, of the reagent added, and of the precipitate produced will always be given; these furnish sufficient data to enable the student to form the equation; only where the equation is exceptionally difficult or complicated will it be given in full.

Abbreviations.—For convenience in hasty writing, it is not uncommon to employ abbreviated formulæ for the compound radicles, especially for such as are constantly met with or are of unusual complexity. In the text only such abbreviations are employed as save the use of brackets or of numbers: thus Am_2 is a short rendering of $(NH_4)_2$, and Ho_6 of H_6O_6.

The following are the abbreviations used:—

Am for (NH_4), the metallic radicle ammonium.
$\bar{T}$ „ $(C_4H_4O_6)$, the acid radicle of tartrates.
$\bar{A}$ „ $(C_2H_3O_2)$, the acid radicle of acetates.
$\overline{Cfy}$ „ $(FeCy_6)$, in ferro- and ferricyanides.
Ho „ (HO), the radicle occuring in hydroxides.

The following contracted words are also used:—

expt.	for	experiment.	*insol.*	for	insoluble.
pp.	„	precipitate.	*sol^n.*	„	solution.
pp^n.	„	precipitation.	*cryst^{ne}.*	„	crystalline.
pp^d.	„	precipitated.	*col^n.*	„	coloration.
sol.	„	soluble.			

The example of entry which follows has been selected from paragraphs (47-50): it will serve as a specimen of the brief entry of reactions in the note-book.

POTASSIUM (K).—Used KCl.

1. *$PtCl_4$*: stirred on watch glass: yellow cryst^{ne} pp., sol. in much water and in KHO, insol. in alcohol: $2KCl+PtCl_4=\overline{K_2PtCl_6}$.
2. *$NaH\bar{T}$*: well shaken in a test-tube: white cryst^{ne} pp., sol. in water, in KHO, and in HCl: $KCl+NaHT=\overline{KH\bar{T}}+NaCl$.
3. *Flame col^n.*: pale lilac, crimson through the indigo-prism; a red and a violet line at the extreme ends of the spectrum.
4. *Heated in tube or on platinum foil:* solid KCl melted, but gave no fumes: hence it is not volatile.

GENERAL RULES FOR WORKING.

44. The student should attend carefully to the following precautions: they will most materially aid the progress and accuracy of his work, and, if observed from the commencement, they will not be felt irksome.

1. Before commencing work look through the reagent bottles belonging to the working bench (550); replace any which are absent, and see that the bottles are arranged in the order shown by the number on the labels. Then proceed to fill any which are empty, and to relabel any whose labels are imperfect or loose: also filter any of the liquid reagents which are not quite clear when shaken.

2. Arrange the apparatus required for use on the back part of the bench, so as to leave the front part free to work upon; this will also lessen the risk of upsetting anything. See Frontispiece.

3. All glass and porcelain which is not in use should then be carefully cleansed; but this ought to be unnecessary, as apparatus should never be put away dirty.

4. When using a reagent bottle take it in the right hand, remove the stopper by grasping it between two of the fingers, or between the fourth finger and palm, of the left hand; hold the stopper in this way, and replace it immediately after the bottle has been used. Pour the liquid out of the side opposite to that bearing the label, and prevent the last drop from running down the outside by touching the lip of the bottle either with the stopper or against the clean edge of the vessel. Solid reagents should be taken out of the bottle on a clean knife-blade or horn spatula, or with a slip of clean cardboard or paper. *Each bottle must be restoppered and replaced on the shelf in its proper place with the label outwards immediately after use, and must never be left standing on the working bench.*

5. If a solution or precipitate has to be put by for some time always label it with a piece of gummed paper bearing an inscription, or a reference number which is explained in the note-book, and which is sufficient to recall to mind what the

solution or precipitate consists of: *never trust to memory in this matter.*

6. Brass crucible tongs must never be employed for holding hot evaporating basins containing acids, as some of the brass is apt to be dissolved and thus introduced into the solution. A hot dish may be moved either by holding it with a cloth or by lifting the stand upon which the dish is placed.

7. *Liquids only* are to be poured down the sinks; all *solid* refuse, such as soiled filters, broken glass, and pieces of charcoal, must be thrown into a box placed in an accessible position in the laboratory, or into a small earthenware jar or dish placed upon each working bench.

8. When a substance is being examined to find out its composition the student must fully write down in the notebook each test or process, with the result obtained, *as soon as it is completed.* The analysis is thus written out gradually as it progresses: *on no account must the entry of the results be left until the completion of the analysis.* This rule cannot be too strongly enforced, as neglect of it always causes uncertainty and confusion. The most convenient form of entry is that of the Analytical Tables.

9. *Cleanliness and neatness* in analytical work cannot be too carefully attended to: more confusion and error may be caused by using dirty test-tubes, beakers, dishes, and funnels than would be thought possible by the beginner. Work should also be rendered as *systematic* as possible, in order to economise time and thought.

45. *The following directions for trying the analytical reactions* are important:—

1. A *solution* of the substance, the reactions of which are to be tried, must always be employed, unless it is specially stated that the *solid* is required. Solid substances are usually necessary only for blowpipe reactions. A solution may be kept in readiness, or it can be easily made from the solid by warming it with water or in some cases with dilute acid (553–4). This liquid is often called *the original solution.*

2. Take in a test-tube or small beaker about twenty cubic centimetres of the solution of the substance. If the solid substance is also required, take some of it upon a watch glass and powder it finely.

3. Pour a little of the solution into a perfectly clean test-tube and add the first-mentioned reagent to it. A few drops of the reagent should be added at first; more can be employed afterwards if excess is required. The student must from the first acquire the habit of working with small quantities both of solutions and reagents. In trying each of the reactions a separate portion of the original solution is to be taken.

4. Before trying a reaction, the student should carefully ead through the whole of the description of the results to be obtained; the experiment is then to be performed, literally following out the directions given. It is a necessary condition of after success that each result should be obtained precisely as is stated in the text; and the student must never on any account pass on without being satisfied that the statements of the book are true, and that he could at any time repeat the test successfully.

5. The water used for the tests and reactions must always be distilled water. The acids are to be used in a dilute condition unless it is stated that the strong acid is to be employed. It will always be specified when solid reagents are to be used, otherwise they are to be employed in solution. In all cases KHO and NaHO may be used indifferently the one for the other.

REACTIONS FOR THE METALS.

In trying through the reactions for the metals the usual order of the analytical groups has been reversed, because the most simple reactions occur in the last groups and they are therefore better suited for a beginner.

GROUP V.—THE POTASSIUM GROUP.

46. This group includes K, NH_4, Na, Mg: the first three of these belong to the group of the *alkali metals.*

The members of this group are not precipitated by any group reagent.

Potassium (K).—Use KCl.

47. *$PtCl_4$*, if added to some of the KCl solution on a watch glass and stirred gently but persistently with a glass rod, gives a yellow crystalline precipitate (K_2PtCl_6), which forms first along the lines where the rod has rubbed the glass.

Stir up the precipitate and pour off the liquid with the precipitate into three test-tubes; to one add much distilled water and heat, the precipitate dissolves: to another part add alcohol, the precipitate does not dissolve. Hence this precipitate is soluble in water, and insoluble in alcohol. By warming the third portion with solution of KHO, the precipitate is shown to be soluble in that liquid.

Since this precipitate is soluble in alkalis, the liquid should always be neutral or faintly acid before adding $PtCl_4$; it is always well to make sure by test paper that the liquid is slightly acid in reaction (34). Further, as the precipitate is more insoluble in alcohol than in water, addition of alcohol often hastens its formation in dilute solutions. Owing to the solubility of the precipitate in water, very dilute solutions must be evaporated nearly to dryness and cooled before applying the test.

48. *$NaH\bar{T}$*, if added to some of the KCl solution in a test-tube, gives a white crystalline precipitate ($KH\bar{T}$); this precipitate usually appears only when the mouth of the test-tube is closed with the thumb and the tube is well shaken for some time.

Shake up the precipitate and pour it off with the liquid into four test-tubes; add to these different portions, water, KHO, and HCl respectively; on being shaken or warmed with each of these liquids, the precipitate will dissolve. To the fourth portion add alcohol; the precipitate does not dissolve.

In using the above test for the detection of potassium, the precipitate will not be obtained at once in dilute solutions; but its formation is much hastened by the addition of alcohol; very dilute solutions must first be concentrated by evaporation. The solution to be tested should be neutral or feebly alkaline: if it is strongly acid in reaction, the free acid should be neutralised by the addition of Na_2CO_3 solution, or if the acid is volatile it may be removed by evaporation; feebly acid solutions may be at once mixed with $NaH\bar{T}$; to an alkaline solution $H_2\bar{T}$ should be added until the reaction of the liquid is strongly acid.

49. *Flame coloration* (41).—Dip a loop of platinum wire, which gives no colour to the flame, into some strong KCl solution, or moisten it and dip it into some finely powdered KCl, and hold the loop in the flame. A *pale lilac* coloration will be seen if the KCl is pure; but whatever tint is thus seen, the flame will always appear deep *crimson red* through the thicker parts of an indigo prism.

The flame coloration due to potassium shows in the spectroscope a *red* line (α) and a faint *indigo blue* line (β) at the extreme ends of the spectrum (see page 66); a faint continuous spectrum is also visible.

50. *Heat a little solid KCl* in a small dry test-tube, or powder it finely and heat it on a piece of platinum foil; the KCl will melt, but it will not pass into vapour and produce white fumes unless it is heated very strongly, since potassium salts are only slightly volatile even at a bright red heat. The fused residue is often transparent and invisible; its presence is detected, however, by a slight crackling being heard during the cooling of the foil.

AMMONIUM (NH_4).—Use NH_4Cl.

51. *KHO*, if poured into some of the solution, or upon the solid, and heated, gives off ammonia gas,

$$NH_4Cl + KHO = NH_3 + KCl + H_2O$$

which is recognised by the following properties.

a. By its pungent smell, which is that of common smelling salts.

b. By turning moistened red litmus paper blue, or turmeric paper brown: wet a small piece of the test paper with distilled water, and place it upon the end of a glass rod; then hold the rod in the test-tube, taking great care not to bring it into contact with the liquid or with the sides of the tube; the change of colour will take place.

c. By yielding white fumes with an acid gas or vapour. Hence if a glass rod or stopper, wetted with strong HCl, HNO_3, or $H\bar{A}$, is held over the mouth of the test-tube, dense white fumes will be seen.

52. *$PtCl_4$* when stirred on a watch glass with some AmCl solution gives a yellow crystalline precipitate (Am_2PtCl_6); this precipitate is soluble in water and in KHO, but insoluble in alcohol. If it is boiled with KHO, it evolves NH_3, differing in this way from K_2PtCl_6. The remarks at the end of (47) apply to this reaction also.

53. *$NaH\bar{T}$*, when shaken for some time in a test-tube with AmCl solution, gives a white crystalline precipitate ($AmH\bar{T}$); which is soluble in H_2O, in KHO, and in HCl, but is insoluble in alcohol. The remarks at the end of (48) apply also to this reaction.

54. *Flame coloration.*—Pure AmCl gives no flame coloration: but it almost invariably contains a trace of sodium, which imparts a yellow colour to the flame.

55. *Heat a little solid AmCl* on a piece of platinum foil, it will be converted into vapour and will produce white fumes; these are best seen if the foil is removed for a moment from the flame: the solid at last entirely disappears, showing that AmCl is completely volatile.

Repeat the experiment by heating a small piece of solid AmCl in a dry test-tube; the solid is completely volatilised, and is condensed as a white coating or *sublimate* on the upper part of the tube.

SODIUM (Na).—Use NaCl.

56. *Flame coloration.*—NaCl gives an *intense yellow* colour to the flame; the colour is either invisible or appears only pale blue when viewed through the indigo prism, *and never shows the slightest tinge of red* when seen through the thicker parts of the prism. A crystal of red potassium dichromate appears colourless if illuminated by this yellow flame. Examined by a small spectroscope, the sodium coloration gives a single yellow line *a*, see page 66.

The yellow coloration of the flame due to sodium is always readily seen, since it overpowers the potassium coloration; its production is the most delicate and trustworthy, and often the only possible, test for sodium.

If both potassium and sodium are present, the potassium coloration is unseen, and the yellow coloration of sodium is alone visible; but on examining this flame through the indigo prism, the crimson coloration of potassium is at once seen. Prove this by mixing together KCl and NaCl solutions and examining the flame coloration both with and without the indigo prism. Look at this coloration also through the spectroscope; the lines of Na and K will be distinctly separated from one another.

57. *Heat a little solid NaCl* in a dry test-tube or on platinum foil; it will melt without producing white fumes unless it is heated very strongly, since NaCl is only slightly volatile at a bright red heat.

MAGNESIUM (Mg).—Use $MgSO_4.7H_2O$.

The $7H_2O$ represents the *water of crystallisation* always present in the solid state in the crystallised salt; water of crystallisation is entered in this way throughout the book.

58. *Na_2HPO_4*, added after AmCl and AmHO, gives a white crystalline precipitate ($MgAmPO_4$); if much water is present, this precipitate forms only when the liquid is warmed and well stirred or shaken: it is soluble in HCl and in other acids, but is insoluble in AmHO:—

$$MgSO_4 + AmHO + Na_2HPO_4 = MgAmPO_4 + Na_2SO_4 + H_2O.$$

59. *KHO* gives a white precipitate (MgH_2O_2), readily soluble in acids.

60. *AmHO* gives a white precipitate (MgH_2O_2); but if AmCl is added before the AmHO, no precipitate is produced.

61. *Am_2CO_3* gives a white precipitate ($MgCO_3$) only in strong solutions and on standing for some time: AmCl prevents the formation of this precipitate also.

62. *Flame coloration.*—Pure $MgSO_4$ solution gives no colour to the flame.

63. *Blowpipe test.*—If a little solid $MgSO_4.7H_2O$ is placed in a small cavity on a piece of wood charcoal, and then heated in the tip of the outer blowpipe flame (39), it shines brightly and gives a white unmelted mass of MgO; no white fumes are given off since MgO is not volatile. If this white mass after cooling is moistened with several drops of cobalt nitrate solution and is again heated to whiteness for some time in the outer blowpipe flame, it assumes a *delicate pink* colour: this colour is best seen when the mass is *quite cold* by comparison with a piece of white paper held near the residue on the charcoal.

DETECTION OF ONE METAL IN GROUP V.

64. After having carefully worked through the reactions for the metals in the fifth group, the student should proceed to apply the experience thus acquired to the detection of one metal belonging to this group. A salt containing only one of these metals having been supplied, separate portions of it should be subjected to the tests given in the first column of the following Table of Differences (65).

A glance at this Table will show that those tests have been selected which best serve to distinguish the metals of this group one from another. The order in which these tests are placed is also such as will best serve this object. It will be evident that it is never necessary to try the whole of the tests upon any one substance; the presence of the metal detected by one test may be confirmed by another selected for that purpose. The third and fourth tests are also manifestly alternative ones, the use of $NaH\bar{T}$ being preferable to that of $PtCl_4$ because it is cheaper. A selection is made from the first four tests in examining a liquid: a solid substance would be first subjected to the fifth and sixth tests, a portion of the solid dissolved in water being afterwards examined by the earlier ones.

As each test is made the result yielded by it is carefully observed: occasionally no change is noticed, as is indicated by a blank in the column to the right: if any result is noted it is looked for in the columns to the right, and will indicate the presence of that metal which heads the column in which the result stands.

Sodium is rarely entirely absent from any substance: the quantity present may be judged by the intensity of the *yellow* coloration: unless this is very intense, the presence of a trace of sodium should be noted down, and another metal should be looked for.

The student will naturally take advantage of the aid afforded by the spectroscope in examining the flame coloration (42).

The following example of the entry of a simple analysis will illustrate both the method of selecting the tests and of entering the work done:—

A solid white substance was given to be tested for one metal in Group V.

Test.	Observation.	Inference.
1. Heated in an ignition tube.	Substance fused, but did not volatilise.	Absence of NH_4.
2. Heated on charcoal in the blowpipe flame.	Substance fused, and sank into the charcoal.	Presence of K or Na.
Dissolved part of the substance in water.		
3. Held a drop on a platinum wire loop in the flame.	A pale lilac flame coloration, which appeared crimson through the indigo prism.	Presence of K.
4. Stirred a little of the solution with a few drops of $PtCl_4$ on a watch glass.	A yellow precipitate formed.	Presence of K.
		Found K.

Several simple unknown salts of the metals of Group V. should be examined in this way by the Table (65), and the results carefully entered in the note-book.

65. Group V.—Table of Differences.

Tests.	K—salts.	NH_4—salts.	Na—salts.	Mg—salts.
For solutions.				
1. Boil with KHO solution:	—	NH_3 gas given off: known by its smell and by turning moist red litmus blue; it also gives dense white fumes with strong HCl.	—	White precipitate. Confirm by adding AmCl, AmHO, and Na_2HPO_4, to a fresh part of the solution (58).
2. Heat in the Bunsen flame on a clean platinum wire:	*Pale lilac flame*, which appears crimson through the indigo-prism.	—	*Intense yellow* flame, which shows no tinge of red through the indigo-prism.	—
3. Add $PtCl_4$ make slightly acid with dilute HCl, and stir vigorously:	Yellow crystalline precipitate, forms only in strong solutions.	Yellow crystalline precipitate, forms only in strong solutions.	—	—
4. Add NaH$\overline{T}$ and shake well:	White crystalline precipitate in strong solutions.	White crystalline precipitate in strong solutions.	—	—
For solids only.				
5. Heat to redness in a dry test-tube, or on platinum foil:	Non-volatile, unless heated to bright redness.	Volatile; white fumes are given off and the substance passes into vapour, at least partially, forming a sublimate on the upper part of the tube.	Non-volatile, unless heated to bright redness.	Non-volatile.
6. Heat strongly on charcoal in the outer blowpipe flame:	Easily fusible, sinking into the charcoal, and colouring the flame pale violet. (See 2.)	Volatile, giving off white fumes.	Easily fusible, sinking into the charcoal, and colouring the flame intense yellow. (See 2.)	White infusible residue is left, which if moistened with $Co(NO_3)_2$ solution and reheated becomes pink.

DETECTION OF METALS IN GROUP V.

Paragraphs, the numbers of which are marked with an asterisk, may be omitted by students who are learning the analysis of simple salts only.

66.* When more than one metal of the fifth group is present in the substance to be examined, the possibility of interference in the reactions arises; it should be remembered, however, that—

1. NH_4 may be always detected by the evolution of NH_3 on boiling the substance with KHO solution.

2. Na can always be found by its yellow flame coloration.

3. Mg is indicated with certainty by its precipitation by KHO solution, and by Na_2HPO_4 in the presence of AmHO and AmCl.

4. K may always be found by a crimson flame coloration seen through the indigo prism, or by the red line in its spectrum.

5. The test for K by means of $PtCl_4$ or $NaH\bar{T}$ is only conclusive in the absence of NH_4. If NH_4 is present, therefore, its salt must be removed by ignition before K can be tested for by precipitation.

The student should now proceed to examine by the Table (67) several solutions containing two or more metals of the fifth group. The composition of each solution should be unknown to him, and by trying the tests in the order given in the Table upon separate portions of the solution, the metals present may be discovered. Only one of the observations recorded against each test will be made; in future tables the bracketted paragraphs, which concern the absence of the metals, will be omitted.

The flame coloration obtained in Exp. 2 of this Table should be examined by the spectroscope.

67.* TABLE FOR TESTING FOR Mg, K, Na, NH_4, IN A SOLUTION WHICH MAY CONTAIN ALL OF THEM.

Experiment.	Observation.	Inference.
I. Add to a small part of the solution AmCl, then AmHO and Na_2HPO_4, shake well and allow to stand if no precipitate appears at once.	1. A white precipitate forms.	Presence of Mg.
	[2. No precipitate appears.]	[Absence of Mg.]
II. Dip a small loop of clean platinum wire into the solution, and hold it near the top of the Bunsen flame.	1. A yellow flame.	Presence of Na, possibly also of K.
	Look at the yellow flame through the indigo-prism; a crimson red colour is seen.	Presence of K.
	2. A pale violet flame, appearing crimson red when seen through the indigo-prism.	Presence of K and absence of Na.
	[3. No colour is imparted to the flame.]	[Absence of K and Na.]
III. Boil some of the solution in a test-tube with KHO solution, and try whether NH_3 is given off by smelling; or if it cannot be smelt, by testing with moistened red litmus-paper or strong HCl.	1. NH_3 gas is smelt, or the red litmus becomes blue, and white fumes are obtained with HCl.	Presence of NH_4.
	[2. No NH_3 is smelt, and moist red litmus paper is not blued, neither are white fumes obtained with HCl.]	[Absence of NH_4.]

IV. A confirmatory test for K may be made by adding $PtCl_4$ or $NaH\overline{T}$. If Exp. III. has shown that NH_4 is present, proceed to test for K by column A; if NH_4 is absent, test for K by column B (66, 5).

A.	B.
Boil some more of the solution down to dryness in a porcelain dish; scrape out the white substance left in the dish, and heat it strongly on a piece of platinum-foil as long as any white fumes are seen to be given off when the foil is taken out of the flame for an instant. Place the foil when cold in a test-tube, and boil it with *very little* water, to which a drop of dilute HCl has been added, and evaporate to several drops; cool the liquid, and pour it out upon a watch glass, add a little $PtCl_4$, and stir well:— A yellow precipitate:—*Presence of K.*	Pour a little of the solution upon a watch glass, add $PtCl_4$, and stir well: a yellow precipitate:—*Presence of K.* If no precipitate forms, K may nevertheless be present, the solution being too dilute to allow the precipitate to form; therefore pour some of the solution into a porcelain dish, boil down nearly to dryness, cool and test this liquid by stirring it with $PtCl_4$ on a watch glass, adding alcohol if the precipitate does not appear at once: a yellow precipitate:—*Presence of K.* *Note.*—Addition of alcohol will frequently produce the precipitate without evaporation.

GROUP IV.—BARIUM GROUP.

68. This group includes Ba, Sr, Ca.

These metals differ from those of Group V. by being precipitated as carbonates by Am_2CO_3, even in the presence of AmCl. They are not precipitated by any other group reagent. The carbonates are soluble in boiling solution of AmCl.

BARIUM (Ba).—Use $BaCl_2.2H_2O$.

69. *Am_2CO_3* added after AmCl causes a white precipitate ($BaCO_3$), which is at first flocculent, but slowly shrinks in bulk and becomes crystalline if it is gently heated and allowed to stand: prove that this precipitate is soluble in $H\bar{A}$.

70. *$CaSO_4$, or H_2SO_4:* heavy white precipitate ($BaSO_4$), insoluble in HCl even on boiling.

71. *K_2CrO_4* added after some $H\bar{A}$: yellow precipitate ($BaCrO_4$), which is soluble in warm HCl, but differs from $SrCrO_4$ by being insoluble in warm $H\bar{A}$.

72. *H_2SiF_6:* semi-transparent precipitate ($BaSiF_6$): the precipitate does not appear in dilute solutions until after some time, or until the liquid is boiled, or alcohol is added: shaking or stirring the liquid hastens its formation. H_2SiF_6 yields no precipitates with solutions of Sr and Ca salts.

73. *$Am_2C_2O_4$:* white precipitate (BaC_2O_4).

74. *Flame coloration:* $BaCl_2$ gives a yellowish-green colour to the flame, visible through the indigo prism.

The spectrum (p. 66) consists of a number of lines, the most characteristic of which are three green lines, α, β, γ.

STRONTIUM (Sr).—Use $Sr(NO_3)_2.4H_2O$.

75. *Am_2CO_3* added after some AmCl: white precipitate ($SrCO_3$), soluble in $H\bar{A}$; this precipitate is flocculent at first, but quickly becomes crystalline and shrinks very considerably when heated.

76. *$CaSO_4$, or H_2SO_4:* white precipitate ($SrSO_4$): this precipitate does not usually form in a cold solution at once, but only after standing for some time: the precipitate, however, *appears at once on boiling the liquid.*

77. *K_2CrO_4* added after some $H\bar{A}$: no precipitate, since $SrCrO_4$ is soluble in $H\bar{A}$.

78. *$Am_2C_2O_4$:* white precipitate (SrC_2O_4).

79. *Flame coloration:* crimson red; this colour appears *intense red* through the indigo prism unless the flame coloration is very faint.

The strontium spectrum contains many lines; the most characteristic are the orange line (α), the red lines (β, γ) and the blue line (δ).

CALCIUM (Ca).—Use $CaCl_2.6H_2O$.

80. *Am_2CO_3* added after AmCl: white precipitate ($CaCO_3$), soluble in $H\bar{A}$: this precipitate is flocculent at first, but shrinks considerably and becomes crystalline after standing for some time, or when it is gently heated.

81. *$CaSO_4$:* no precipitate, even when the liquid is allowed to stand, or is boiled.

82. *H_2SO_4:* a white precipitate ($CaSO_4$) forms at once in strong solutions, and often in weak solutions when they are boiled; but some $CaSO_4$ will always remain dissolved, since it is not quite insoluble in water; prove this by boiling the liquid containing the precipitate and filtering it; then keep adding AmHO to the filtrate and stirring it until a drop of the solution turns red litmus paper blue and the liquid smells of NH_3; then add $Am_2C_2O_4$, a white precipitate will form, showing the presence of Ca (85) which must have been dissolved as $CaSO_4$.

83. *K_2CrO_4:* no precipitate.

85. *$Am_2C_2O_4$:* white precipitate (CaC_2O_4), soluble in most acids, but insoluble in $H\bar{A}$ and in $H_2C_2O_4$.

86. *Flame coloration:* yellowish-red; this colour viewed through the indigo prism *does not appear red* but *dingy green*, thus differing from that given by Sr.

In the calcium spectrum the most characteristic lines are the green line (β) and the intense orange line (α).

87. GROUP IV.—TABLE OF DIFFERENCES.

The first three tests only need be used for the detection of a member of this group occurring by itself in solution: test 1 serves also for solids. The flame coloration obtained in Exp. 1 should be examined by the spectroscope.

Tests.	Ba—salts.	Sr—salts	Ca—salts.
1. Flame coloration:	Greenish-yellow.	Intense crimson, appears *crimson* through the indigo-prism.	Yellowish red, appears *dingy green* through the indigo-prism.
2. $CaSO_4$ added to the cold solution:	An immediate precipitate.	A precipitate appearing only after a short time *or immediately on boiling.*	——
3. K_2CrO_4 added after $H\bar{A}$:	Light yellow precipitate.	——	——
[4. H_2SiF_6 added:	White precipitate.	——	——]
5. H_2SO_4 added in excess and boiled:	*Entirely* precipitated as $BaSO_4$.	*Entirely* precipitated as $SrSO_4$.	*Partially* precipitated as $CaSO_4$ (82).

SEPARATION AND DETECTION OF METALS IN GROUP IV.

88.* The following differences are made use of:—

1. The different flame colorations yielded by these metals, examined if necessary through the indigo prism, or by means of the spectroscope.

2. The difference in the behaviour of their solutions with $CaSO_4$ solution, which at once indicates the presence or absence of Ba, or of Sr in the absence of Ba.

3. The insolubility of $BaCrO_4$ in $H\bar{A}$, and hence the possibility of separating Ba, if present, by adding $H\bar{A}$ and then K_2CrO_4.

4. The fact that Sr but not Ca can be precipitated by $CaSO_4$ on boiling; this reagent will therefore indicate the presence of Sr in the absence, or after the separation, of Ba.

5. The complete precipitation of Sr, if present, by boiling the solution with H_2SO_4; sufficient $CaSO_4$ still remaining in solution to give in the filtrate made alkaline with AmHO a precipitate of CaC_2O_4 on addition of $Am_2C_2O_4$.

89.* *A solution which has to be examined for Ba, Sr, and Ca,* and can contain only these substances, is made alkaline, if it is not already so, by the addition of AmHO; Am_2CO_3 is then added as long as it causes any further precipitate after the liquid has been stirred well and allowed to settle: the liquid is then filtered: a little more Am_2CO_3 is added to the clear filtrate, and if this causes any furthur precipitate more Am_2CO_3 is added, and the liquid is again poured through the same filter.

As soon as the filtrate gives no further precipitate with Am_2CO_3, all the members of this group which were present in the solution will have been precipitated as carbonates, and on filtration will be obtained on the filter. The precipitate is then examined by Table IV. (438).

GROUP III.A.—IRON GROUP.

90. This group includes Al, Fe, Cr.

The members of this group differ from those of Groups III.B, IV., and V. by being precipitated by AmHO after addition of AmCl; they are not precipitated however by the group-reagents for Groups II. and I. This group is also precipitated by Am_2S, or by H_2S added to the alkaline solution. Group III.A. further differs from Group III.B. in being completely precipitated by adding $BaCO_3$ shaken up with water; this reagent affords the most perfect means of separating these two sub-groups. The members of this group show no characteristic flame colorations; but, with the exception of Al, they impart a characteristic colour to the borax bead.

ALUMINIUM (Al).—Use $AlAm(SO_4)_2.12H_2O$.

91. *AmHO:* white flocculent precipitate ($Al_2H_6O_6$), which is seen best on boiling the liquid: it is soluble in HCl, in $H\bar{A}$, and slightly soluble in AmHO in the absence of AmCl:—$2AlAm(SO_4)_2 + 6AmHO = Al_2H_6O_6 + 4Am_2SO_4$.

92. KHO added in very small quantity: white flocculent precipitate ($Al_2H_6O_6$), soluble in excess of KHO. If to a part of the KHO solution HCl is added gradually until it is neutral, the $Al_2H_6O_6$ is reprecipitated; if more acid is added the precipitate dissolves, but reappears on adding AmHO in slight excess. From another portion of the KHO solution the $Al_2H_6O_6$ may be reprecipitated by the addition of sufficient AmCl solution.

93. *Am_2S:* same precipitate ($Al_2H_6O_6$), H_2S gas being evolved or remaining dissolved in the liquid:—

$$2AlAm(SO_4)_2 + 3Am_2S + 6H_2O = Al_2H_6O_6 + 4Am_2SO_4 + 3H_2S.$$

94. *Blowpipe reaction.*—Solid $AlAm(SO_4)_2.12H_2O$ when heated on charcoal in the outer blowpipe flame, and then moistened, after cooling, with $Co(NO_3)_2$ solution and again heated in the outer flame, gives a *fine blue mass.*

IRON (Fe).—Use Fe_2Cl_6, and $FeSO_4.7H_2O$.

95. Two classes of iron compounds are known, which differ in appearance and properties and behave differently with reagents: they are distinguished as *ferrous* and *ferric* compounds respectively; in the former class the metal may be distinguished by the name *ferrosum* and by the symbol (Fe″), and in the latter by *ferricum* (Fe‴). The dashes following the symbol denote the number of Cl atoms, or their equivalent, which are combined with one atom of Fe in its compounds.

It is usual in stating analytical results to mention in which state of combination the iron exists, and therefore some distinctive tests are described below (101). The whole of the reactions of ferrous compounds are not given, because Fe is always separated and detected in the course of analysis as a ferric compound, and ferrous compounds are found by special reactions tried on the original substance.

Ferrous are readily converted into ferric compounds by boiling them for a short time with a little strong HNO_3, or with HCl and a crystal of $KClO_3$.

Both classes of compounds give the same results in the blowpipe reactions (99,100).

Ferric salts are usually yellow or reddish yellow; ferrous salts are usually pale green, or white if perfectly anhydrous.

Use Fe_2Cl_6 solution for reactions (96–98); and solid $FeSO_4.7H_2O$ for (99, 100). In preparing $FeSO_4$ solution for (101), crush a crystal of the solid substance and shake it in a test-tube with *cold* water.

96. *AmHO, or KHO:* reddish-brown flocculent precipitate ($Fe_2H_6O_6$): insoluble in KHO, soluble in HCl.

97. *Am_2S:* a black precipitate (FeS), mixed with white sulphur: FeS is soluble in boiling acids, but insoluble in KHO:—$Fe_2Cl_6 + 3Am_2S = \overline{2FeS + S} + 6AmCl$.

In a very dilute iron solution a green colour only is produced at first by Am_2S, but black FeS separates after the liquid has stood for some time.

98. *H_2S* gas passed into the solution, or added as H_2S-water: black precipitate (FeS) in alkaline solutions: in neutral or acid solutions sulphur is precipitated and the ferric is changed into a ferrous salt; the colour of the solution therefore changes from yellow to pale green as is seen after boiling and filtering a strong solution:

$$Fe_2Cl_6 + H_2S = 2FeCl_2 + 2HCl + S.$$

99. *Fused with Na_2CO_3 on charcoal* in the inner blowpipe flame, a solid iron compound leaves a grey powder; this is shown to be *magnetic* by being attracted when touched under water with the end of a magnet, or with the point of a magnetised knife-blade (39, 40).

100. *A borax bead* (38) containing Fe, is *reddish-brown* while hot, and *yellow* when cold, after having been fused in the outer blowpipe flame: after fusion in the inner flame it is *olive green*.

101. DISTINCTIVE TESTS FOR FERROUS AND FERRIC SALTS.

For these tests several drops of dilute HCl should be first added to the Fe solution.

Reagents to be added.	Ferric-salts. (Use Fe_2Cl_6 solution.)	Ferrous-salts. (Use $FeSO_4$ solution.)
1. K_4FeCy_6 added in small quantity:	*Dark-blue precipitate*, "Prussian blue"; insoluble in HCl *, soluble in $H_2C_2O_4$, and slightly soluble in K_4FeCy_6 added in excess; turned brown by KHO.	*Light-blue precipitate*, becoming dark-blue in the air, or on addition of HNO_3 or Br-water.
2. $K_6(FeCy_6)_2$:	*No precipitate:* the solution darkens, but on dilution with water is seen to contain no precipitate.	*Dark-blue precipitate*, "Turnbull's blue": insoluble in HCl. In *very* dilute solution only a dark bluish grey colour is produced.
3. *KCyS*:	*Blood-red coloration:* no precipitate is produced, the liquid being perfectly clear on dilution; the colour is immedidiately destroyed when several drops are poured into some $HgCl_2$ solution: its production is hindered by HNO_3 and by $H\overline{A}$.	*No red coloration* unless small quantities of ferric-salts are present.
4. *KHO*:	*Reddish-brown* precipitate.	*Dingy-green* precipitate, turning brown in the air.

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* The solubility and insolubility of this precipitate must be shown by warming separate portions of the blue liquid containing the precipitate with HCl, $H_2C_2O_4$ and K_4FeCy_6, then separately filtering each: if the precipitate has been dissolved the filtrate will be dark blue.

CHROMIUM (Cr).—Use $CrK(SO_4)_2.12H_2O$.

Chromic salts are usually bluish-green or violet in colour.

102. *AmHO:* pale bluish-green or purple precipitate ($Cr_2H_6O_6$): if AmHO is added in large quantity and the liquid is heated, some of the precipitate dissolves, producing a beautiful violet-red solution: the colour is best seen after filtering off the precipitate: from this solution the $Cr_2H_6O_6$ is reprecipitated on boiling the liquid for several minutes in a porcelain dish.

103. *KHO*, if added in small quantity, gives the same precipitate ($Cr_2H_6O_6$): if more *cold* KHO is added, the precipitate is entirely dissolved to a green fluid; on diluting this liquid with water and boiling for several minutes, the $Cr_2H_6O_6$ is reprecipitated and the liquid becomes colourless: AmCl added to the KHO solution also reprecipitates the $Cr_2H_6O_6$.

If to some of the green liquid, obtained by adding KHO in excess to the chromium solution, a little red lead or lead peroxide (PbO_2) is added and the liquid is then boiled, a yellow liquid is obtained, from which a yellow precipitate ($PbCrO_4$) is thrown down on the addition of $H\bar{A}$ in excess.

104. *Am_2S* also precipitates $Cr_2H_6O_6$, H_2S gas being given off or remaining dissolved. See the reaction in paragraph (93).

105. *Fusion of a solid substance containing Cr* with Na_2CO_3 and KNO_3 on platinum or porcelain yields a mass which is coloured yellow by Na_2CrO_4. On dissolving this in boiling water a yellow solution is obtained, which, if acidified with $H\bar{A}$ and boiled for a few minutes to drive off CO_2, gives with $Pb\bar{A}_2$ solution a yellow precipitate of $PbCrO_4$.

106. A *borax bead* containing Cr is green after having been fused either in the outer or in the inner blowpipe flame.

Cr forms two classes of compounds: in one class Cr in combination with oxygen acts as an acid-radicle: this class includes the chromates such as K_2CrO_4 and $K_2Cr_2O_7$, they are usually yellow or reddish in colour; but Cr also forms a series of salts in which it acts as a metallic radicle; these are usually green or violet and give the above reactions for Cr. The latter compounds pass by oxidation into the former in reactions (103, 105), where the oxidation is caused by PbO_2 and by KNO_3 respectively. Chromates pass by reduction into green chromic compounds; examples of this change will be found under the tests for chromic acid (286).

107. Group III.A.—Table of Differences.

Tests.	Al—salts.	Fe'''—salts.	Fe''—salts.	Cr—salts.
For liquids. 1. Add AmHO:	*White* flocculent precipitate.	*Reddish brown* flocculent precipitate.	*Dingy green* precipitate, rapidly turning brown when left exposed to the air.	*Pale green* flocculent precipitate, colour unaltered by exposure to air.
2. Add KHO:	*White* flocculent precipitate, *easily soluble in excess of KHO, not reprecipitated on diluting and boiling for some time.*	*Reddish brown* flocculent precipitate, *insoluble in excess of KHO.*	The same precipitate as with AmHO, *insoluble in excess of KHO.*	*Pale green* precipitate, *soluble in excess of cold KHO, but reprecipitated on diluting and boiling for some time.*
3. Add KCyS and a few drops of HCl:	——	*Deep blood red* coloration, destroyed by pouring into $HgCl_2$ solution.	*No coloration* unless Fe''' is also present.	——
Blowpipe tests for solids. 4. Fuse with Na_2CO_3 and KNO_3 on platinum foil:	——	On cooling, a white mass of Na_2CO_3 remains, with dark brown particles of Fe_2O_3.	Same as Fe'''.	On cooling, a *yellow* mass remains; the solution in $H\bar{A}$, gives a yellow precipitate with $Pb\bar{A}_2$
5. Heated on charcoal:	If moistened with $Co(NO_3)_2$ solution and re-heated strongly gives a *fine blue mass.*	In inner flame mixed with Na_2CO_3 gives a grey magnetic powder.	Same as Fe'''.	——
6. Fused in borax bead:	——	*Reddish yellow* in outer flame. *Greenish yellow* in inner flame.	Same as Fe'''.	*Green* both in outer and inner flames.

SEPARATION AND DETECTION OF METALS IN GROUP III.A.

108.* The following differences are utilised:—

1. The solubility of $Al_2H_6O_6$ in *boiling* KHO, in which $Fe_2H_6O_6$ and $Cr_2H_6O_6$ are insoluble.

2. The conversion of $Cr_2H_6O_6$ by fusion with Na_2CO_3 and KNO_3 into soluble Na_2CrO_4: $Fe_2H_6O_6$ when thus treated remaining as Fe_2O_3, which is insoluble in water.

3. Cr is then detected by the yellow colour of the solution of Na_2CrO_4 in water, and by the formation of a yellow precipitate on acidifying it with $H\bar{A}$ and adding $Pb\bar{A}_2$.

4. Fe is found by the blood red coloration obtained on adding KCyS to the Fe_2O_3 dissolved in HCl.

109.* *A solution which may contain Al, Fe, Cr,* is examined in the following manner:—

To a few drops of the liquid acidified with HCl add a little K_4FeCy_6: if a blue precipitate is produced Fe is present: this portion is rejected.

Two other small portions of the acidified solution are then tested; one with $K_6(FeCy_6)_2$, which by giving a dark blue precipitate would show the presence of Fe″; the other with KCyS, which may cause a blood-red colour proving the presence of Fe‴.

Boil the rest of the solution, adding a few drops of strong HNO_3 if Fe″ is present; add some AmCl, then AmHO until the liquid after being well stirred is alkaline and smells of NH_3; boil, filter, and examine the precipitate by Table III.A (436), disregarding all the appended notes

GROUP III.B.—ZINC GROUP.

110. This group includes Zn, Mn, Ni, Co.

Compounds of these metals differ from those of metals of Groups IV. and V. by being precipitated from their neutral or alkaline solutions by Am_2S and from their alkaline solutions by H_2S; they are not precipitated by the group reagents for Groups III.A, II. and I.; but Mn is precipitated by oxidation, if its solution mixed with AmCl and excess of AmHO is exposed to the air.

The compounds of the metals of this group show no characteristic flame colorations; but with the exception of those of Zn they give characteristic colours to a borax bead.

Finely powdered $BaCO_3$ does not precipitate the compounds of the metals of this group from their solutions as it does those of the metals of Group III.A, unless they are present as sulphates.

ZINC (Zn).—Use $ZnSO_4.7H_2O$.

Zinc salts are usually colourless.

111. *Am_2S:* white precipitate (ZnS): the precipitate often appears yellow from the presence of excess of yellow Am_2S in the liquid: its true colour is seen in the next reaction, or is evident when the precipitate produced by Am_2S is separated by settling or filtration. Prove the solubility of this precipitate as is directed in the next paragraph.

112. *H_2S:* white precipitate (ZnS): the Zn is only partly precipitated from a neutral solution, since H_2SO_4 is separated in which ZnS is soluble:—$ZnSO_4 + H_2S = ZnS + H_2SO_4$. But if NaHO or AmHO is added to neutralise the H_2SO_4 the Zn may be entirely precipitated: addition of sufficient $Na\bar{A}$ solution also causes complete precipitation, since the H_2SO_4 in the solution is thus replaced by $H\bar{A}$, in which ZnS is insoluble:

$$ZnSO_4 + H_2S + 2Na\bar{A} = ZnS + Na_2SO_4 + 2H\bar{A}.$$

Add to some $ZnSO_4$ solution AmCl, then AmHO until it is alkaline, and then H_2S, white ZnS will be precipitated: show by adding to separate portions of this liquid HCl, $H\bar{A}$ and Am_2S, that ZnS is soluble in HCl and insoluble in $H\bar{A}$ and in Am_2S.

The tests which show the solubility of the sulphides of the metals of this group are best tried on the precipitate obtained by adding AmCl, AmHO in excess and H_2S, unless freshly prepared *colourless* Am_2S can be obtained, since acids precipitate sulphur from *yellow* Am_2S.

113. *KHO* added in small quantity gives a white precipitate (ZnH_2O_2): if more KHO is added, this precipitate dissolves, but is formed again on adding much water and boiling: or the Zn may be precipitated as ZnS from the KHO solution by the addition of H_2S.

114. If finely powdered $ZnSO_4.7H_2O$ and Na_2CO_3 are mixed in a small cavity on wood charcoal, and the mixture is then heated in the inner blowpipe flame, an incrustation is obtained on the charcoal which is *yellow* while hot and *white* when cold. It cannot be driven away by the outer blowpipe flame, but is easily removed by the inner flame. If this incrustation when cold is moistened with cobalt nitrate solution and is then strongly heated in the outer blowpipe flame it becomes *green.*

The above changes of colour are more distinctly obtained by heating a little solid ZnO in an ignition tube or in the outer blowpipe flame on charcoal, the oxide is *yellow* while hot and *white* when cold: if it is moistened on charcoal with $Co(NO_3)_2$ solution and then reheated in the outer blowpipe flame, the ZnO becomes *green.*

MANGANESE (Mn).—Use $MnCl_2$, or $MnSO_4$.

Manganous salts are usually of a *pale pink* colour: alkaline manganates are green, permanganates purple.

115. *Am_2S:* flesh coloured or pale pink precipitate (MnS), the colour of which is best seen on filtering it from the yellow Am_2S: the precipitate occasionally becomes dark green after standing for some time; the colour of the moist filtered precipitate always changes by oxidation to dark brown on standing *in the air (118).* For the solubility of this precipitate see (116).

116. *H_2S* precipitates MnS partly from neutral solutions, entirely from alkaline solutions, but not at all in presence of free HCl or $H\bar{A}$: show with separate portions of the MnS precipitate, obtained by adding AmCl, AmHO in excess, and then H_2S, that MnS is soluble in HCl and in $H\bar{A}$, but is insoluble in Am_2S.

117. *KHO:* white precipitate (MnH_2O_2), quickly turning brown by oxidation; this change is most rapid when the precipitate is filtered off: the precipitate is insoluble in excess of KHO.

118. *AmHO* gives the same precipitate; but if sufficient AmCl is first added, AmHO produces no immediate precipitate; the solution, however, on standing exposed to the air, gradually turns brown by oxidation, and the Mn is ultimately precipitated as brown $Mn_2H_6O_6$.

119. Any solid substance containing Mn, when fused in the outer blowpipe flame with a mixture of Na_2CO_3 and a little KNO_3 *upon platinum foil*, yields a *bluish green mass* on cooling: the mixture should be fused by heating the *under surface* of the foil with the tip of the blowpipe flame, and the flame must not come into contact with the mixture.

The test may also be made by fusing a mixture of Na_2CO_3 and KNO_3 into a bead in a loop of platinum wire, and then fusing the substance into this bead by heating it in the extreme tip of the outer flame.

This reaction is very delicate.

119*a*. A solid Mn compound, when fused with Na_2CO_3 on charcoal in the inner blowpipe flame, yields a grey magnetic powder (39, 40).

120. *A borax bead* containing *very little* MnO_2 will be *violet red* whilst hot and *amethyst red* when cool after fusion in the outer blowpipe flame: when heated in the inner flame the bead becomes *colourless.*

This reaction is yielded by minute quantities of manganese compounds.

NICKEL (Ni).—Use $NiSO_4.7H_2O$.

Nickel salts are usually *bright green* in colour.

121. *Am_2S:* black precipitate (NiS); add more *yellow* Am_2S, boil and filter, the filtrate will be coloured brown by NiS dissolved in the excess of Am_2S: pour this filtrate into a porcelain dish and boil it for some time, adding distilled water if necessary to prevent the dish becoming dry; the dissolved NiS will be precipitated, and when filtered off will leave a colourless filtrate. If $H\bar{A}$ is added in excess to the dark filtrate, the dissolved NiS is also precipitated. For the solubility of NiS see the next paragraph.

122. *H_2S:* black precipitate (NiS) in neutral solutions or in solutions containing no free acid except $H\bar{A}$ (112); HCl prevents the precipitation. Add to $NiSO_4$ solution AmCl, AmHO in excess and then H_2S; and show with this precipitate that NiS is insoluble in cold dilute HCl and in $H\bar{A}$, but is soluble when heated with HCl after adding a crystal of $KClO_3$.

123. *KHO:* light green precipitate (NiH_2O_2).

124. *AmHO* added in very small quantity gives a bluish green precipitate (NiH_2O_2), which is soluble in excess of AmHO to a violet blue liquid and is also soluble in AmCl: if AmCl be first added, AmHO causes no precipitate.

125. *KCy* solution, made by dissolving a fragment of the solid in water, when added in small quantity gives a yellowish green precipitate ($NiCy_2$): by the further addition of KCy the precipitate is dissolved, but it is reproduced on the addition of sufficient HCl.

126. Acidify some $NiSO_4$ solution with several drops of $H\bar{A}$; then add KCy solution gradually, whilst stirring or shaking the liquid, until the precipitate at first formed is just redissolved; boil this solution for a short time, then cool it and divide it into two parts: the addition of HCl in *excess to one* part will produce a precipitate of $NiCy_2$, often *only appearing* after a time: and the addition to the other part of Na_2Cl_2O, or of excess of NaHO and then Br-water until the liquid becomes yellow (Na_2Br_2O), will give a black precipitate ($Ni_2H_6O_6$) when the liquid is warmed.

127. A mixture of finely powdered $NiSO_4.7H_2O$ and Na_2CO_3, when fused on charcoal on the inner blowpipe flame, leaves a grey powder which is attracted by the magnet (39, 40).

128. *A borax bead* containing Ni is *violet* or *reddish brown* after fusion in the outer blowpipe flame, and becomes *pale yellow* on cooling; if the bead is heated for some time in the inner flame, it becomes *black* and *opaque.*

Cobalt (Co).—Use $Co(NO_3)_2.6H_2O$.

Cobalt salts are usually *reddish pink* in colour if hydrated, and *blu* when anhydrous.

130. *Am_2S:* black precipitate (CoS); on adding much Am_2S and boiling and filtering, the filtrate is coloured *yellow* by Am_2S and is *not dark coloured,* since CoS unlike NiS is insoluble in Am_2S. For the solubility of CoS see the next paragraph.

131. *H_2S:* black precipitate (CoS), only forming in alkaline solutions or in solutions containing no free acid except HĀ (112); HCl prevents the precipitation. Add AmCl, then AmHO in excess and then H_2S to some $Co(NO_3)_2$ solution, and pour into separate portions of this liquid HCl and HĀ, the precipitate does not dissolve; to the portion containing HCl add a crystal of $KClO_3$ and heat, the precipitate dissolves readily.

132. *KHO:* blue precipitate (CoH_2O_2); the colour of the precipitate changes by oxidation on exposure to the air and when the precipitate is heated.

133. *AmHO:* bluish green precipitate, having the same properties as that produced by KHO; it is soluble in excess of AmHO and in AmCl, and is therefore not formed if AmCl is first added.

134. Freshly prepared *KCy* solution if added in small quantity *gives a reddish* brown precipitate ($CoCy_2$); on adding more KCy solution slowly and whilst shaking the liquid the precipitate dissolves, but it reappears on the addition of HCl.

135. If to some $Co(NO_3)_2$ solution, made acid with a few drops of $H\bar{A}$, KCy solution is added slowly until the precipitate at first formed just redissolves, and the liquid is boiled, then cooled and divided into three parts; it will be found that neither the addition of HCl, nor of Na_2Cl_2O, nor of excess of NaHO solution and then Br-water till the liquid is yellow (Na_2Br_2O), produces any precipitate on warming the liquid.

This difference of behaviour of Ni and Co solutions (126), which have been boiled with excess of KCy, is due to the fact that $NiCy_2$ forms with KCy a compound ($NiCy_2.2KCy$) which is easily decomposed: whereas

139. GROUP III.B.—

Tests.	Zn—salts.	Mn—salts.
For liquids.		
1. Add Am_2S; or better, AmCl, AmHO in excess, and H_2S-water.	*White precipitate*: soluble in cold dilute HCl: *insoluble in $H\bar{A}$.*	*Pink precipitate*: soluble in cold dilute HCl: *soluble in $H\bar{A}$.*
2. Add KHO:	*White precipitate: soluble in excess of KHO.*	*White precipitate*; turning brown in the air: *insoluble in KHO.*
3. Add KCy solution: This test need only be tried when a *black* precipitate has been obtained by Test No. 1.	——	——
Blowpipe tests for solids.		
4. Fused with Na_2CO_3 on charcoal in the inner blowpipe flame.	A white incrustation, which, if moistened with $Co(NO_3)_2$ and heated in the outer flame, turns *green*. The substance itself, when strongly heated after having been moistened with $Co(NO_3)_2$ also becomes green.	A grey powder. The substance, if fused with Na_2CO_3 and KNO_3 on platinum foil in outer flame, gives a *bluish green* mass.
5. Borax bead:	——	{Outer flame.—*Amethyst red.* {Inner flame.—*Colourless.*

$CoCy_2$ forms with KCy the very stable compound $K_6(CoCy_6)_2$, *potassium cobalticyanide.*

136. A solid cobalt compound, when fused with Na_2CO_3 on charcoal in the inner flame, yields a grey magnetic powder (39, 40).

137. *A borax bead* when dipped into a strong solution of $Co(NO_3)_2$ assumes a *fine blue* colour after fusion in either the inner or the outer blowpipe flame. This reaction is yielded by minute quantities of cobalt compounds.

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138. The distinction between Ni and Co in the Table (139) depends on the solubility of NiS in *yellow* Am_2S, which is proved by a dark filtrate being obtained on warming with excess of yellow Am_2S and filtering.

TABLE OF DIFFERENCES.

Ni—salts.	Co—salts.
Black precipitate: soluble in boiling yellow Am_2S to a dark solution: insoluble in cold dilute HCl and in $H\bar{A}$.	*Black precipitate: insoluble in boiling yellow* Am_2S, in cold dilute HCl, and in $H\bar{A}$.
Green precipitate: insoluble in KHO.	*Blue precipitate:* insoluble in KHO.
Added in slight excess to the feebly acid solution and boiled for some time, then Br-water and excess of NaHO added, gives a black precipitate on heating.	Added in slight excess to the feebly acid solution and boiled for some time, then Br-water and excess of NaHO added, gives no precipitate on heating.
A grey magnetic powder.	A grey magnetic powder.
Outer flame. *Violet* or *yellow* while hot. *Yellow* when cold. Inner flame.—*Grey* or *opaque* bead.	Outer and inner flames.—*Fine blue* bead.

SEPARATION AND DETECTION OF METALS IN GROUP III.B.

140.* The methods used depend upon—

1. The solubility of NiS in yellow Am_2S, in which ZnS, MnS and CoS are insoluble: NiS is then precipitated from this solution by boiling it, or by the addition of $H\bar{A}$, and the presence of Ni is confirmed by fusion of the precipitated NiS in a borax bead.

2. The solubility of ZnS and MnS in cold dilute HCl, in which NiS and CoS are almost insoluble.

3. The solubility of ZnH_2O_2 in cold KHO, in which MnH_2O_2 is insoluble; white ZnS is then precipitated from this solution by H_2S, and the undissolved MnH_2O_2 yields a bluish green mass when fused with Na_2CO_3 and KNO_3.

4. The difference of behaviour of the solution of $NiCy_2$ and $CoCy_2$ in KCy with Na_2Cl_2O, or with Na_2Br_2O: and the marked difference in the colour of their borax beads.

Mn may be separated from Zn, Ni and Co by passing H_2S into the neutral solution saturated with $Na\bar{A}$ (112): Mn alone remains in solution (116), the other metals being precipitated as sulphides.

Another method for separating Ni and Co depends upon the fact that whilst Co is precipitated as $Co_2(HO)_6$ from its solution by $BaCO_3$ in the presence of Br, Ni is not precipitated by this means and remains in solution.

141.* *A solution is examined for Zn, Mn, Co, Ni*, by adding AmCl to it, then AmHO until it is alkaline, and then Am_2S until the liquid smells of it after having been well stirred or shaken. The liquid is then heated and a few drops of it are poured upon a filter: if the filtrate is colourless, sufficient Am_2S has not been added; more Am_2S is then to be poured in and the filtrate again examined. As soon as the filtrate is brown or yellow in colour, proving that Am_2S has been added in excess, the whole is heated and filtered and the precipitate is examined by Table III.B (437): the filtrate if yellow is rejected, if brown it is examined for Ni according to (431).

GROUP II.A.—COPPER GROUP.

142. This group includes Hg″, Pb, Bi, Cu, Cd.

The compounds of these metals differ from those of the metals in Groups III.A., III.B., IV. and V. by being precipitated as sulphides by H_2S from their acid solutions: they are not precipitated by HCl or by soluble chlorides, with the exception of Pb, which in strong solution is partially precipitated.

The sulphides differ from those of Group II.B. by being insoluble both in Am_2S and in KHO. If very much acid is present H_2S does not precipitate the members of this group readily or completely, unless their solutions are largely diluted with water.

MERCURICUM (Hg″).—Use $HgCl_2$.

Mercury forms two series of compounds distinguished as *mercuric* and *mercurous:* the metal in these two states behaves differently with reagents, and may be distinctively called *mercuricum* and *mercurosum:* in the latter condition it is classed under Group I. In paragraphs (217, 218) tests are given which serve to distinguish these compounds.

144. *H_2S:* black precipitate (HgS): if the H_2S solution is added slowly the precipitate is first *white,* then *brown* or *orange* and ultimately becomes *black:* these changes of colour during the addition of H_2S are characteristic of mercuric salts.

Filter off the precipitate and wash it free from dissolved chlorides, then place portions of it in three test-tubes: boil one of these with strong HNO_3 and another with strong HCl, the precipitate will not be dissolved by either of these acids: now mix the contents of the two tubes and warm again, the precipitate will dissolve: boil the third portion with Am_2S, the precipitate does not dissolve. Hence HgS is insoluble in hot strong HNO_3, in hot strong HCl, and in Am_2S; but it is easily soluble in a mixture of HNO_3 and HCl, which is *known as aqua regia.*

145. *$SnCl_2$:* white precipitate (Hg_2Cl_2): if more $SnCl_2$ is added the white precipitate becomes grey and consists of fine particles of Hg; if the liquid is boiled and then decanted from the grey precipitate, the Hg particles when heated with strong HCl unite to globules visible through a lens and frequently even to the naked eye:—

$$2HgCl_2 + SnCl_2 = Hg_2Cl_2 + SnCl_4$$
$$Hg_2Cl + {}_2SnCl_2 = 2Hg + SnCl_4.$$

146. *Cu:* if a small strip of copper sheet or a copper coin, the surface of which has been cleansed by rubbing it with sand paper, or by dipping it into strong HNO_3, be immersed in some $HgCl_2$ solution made acid with a drop or two of HCl, it will become coated with a grey film of Hg:— $HgCl_2 + Cu = Hg + CuCl_2$.

If the surface of the Cu, after having been immersed for several minutes, is dried and rubbed hard with a cloth or piece of wash-leather, it will appear more or less whitened like silver, the metal Hg having formed an *amalgam* with the Cu.

The Hg may be readily driven off by heating the Cu strongly; if this heating is performed in a test-tube, or better in a small hard glass tube sealed at one end, a sublimate of globules of Hg will form upon the cool sides of the tube.

147. Mix *a little* finely powdered $HgCl_2$ intimately with about four times as much finely powdered Na_2CO_3, which has been recently dried by strongly heating it in a porcelain dish. Pour this mixture into a small test-tube, which has been perfectly dried by heating it in the flame and sucking out the moist air by a glass tube: then cover the mixture with a layer of Na_2CO_3 dried by ignition.

After perfectly cleansing the inside of the tube heat the layer of Na_2CO_3 strongly, and whilst keeping this hot gradually extend the heat to the mixture: Hg will sublime, condensing in globules on the inside of the tube. Sometimes the globules are so small that the layer appears as a grey film, or they are few and scattered and not easily visible; they may usually be united into larger globules by rubbing the inside of the tube with a glass rod, or the separate globules may be detected by means of a pocket lens: even very minute globules may be seen if they are flattened by pressing them with a glass rod against the inside of the tube.

The powder is most easily placed in the tube by alternately scooping it up with the mouth of the tube and tapping the tube so as to shake the powder to the bottom; or by turning the powder out upon a piece of note-paper folded over into a trough and pouring it thence into the tube, holding both tube and paper over the watch glass so as to catch any substance which may fall.

Both the inside of the tube and the powder must be perfectly dry, else some particles obstinately adhere to the inside of the tube and obscure the sublimate.

The heating must never be commenced until the inside of the tube is perfectly clean: it is cleansed if necessary with a twisted slip of filter paper or with a wooden match. Also if any drops of water condense on the inside of the tube during the first stage of the heating, they must be removed by a twisted piece of filter paper; if however proper care has been taken in drying the power, no moisture will condense. The escape of steam from the powder is very undesirable, as it is very likely to sweep mercury vapour out of the tube.

148. Heat a little solid $HgCl_2$ in a tube closed at one end; the substance sublimes in white fumes, showing that mercury compounds are volatile.

LEAD (Pb).—Use $Pb\bar{A}_2.3H_2O$ in dilute $H\bar{A}$.

149. *H_2S:* black precipitate (PbS): this precipitate is often *red* if much hydrochloric acid is present in the solution, but it becomes black on diluting with water and passing H_2S, or on adding H_2S-water. Filter or decant and show with separate portions of the precipitate that PbS is insoluble in KHO and in Am_2S, soluble in boiling dilute HNO_3, and is converted by boiling strong HNO_3 into insoluble $PbSO_4$.

150. *HCl:* white precipitate ($PbCl_2$), forming only in cold and strong solutions: on boiling the precipitate with sufficient water it dissolves, but the $PbCl_2$ separates again in beautiful crystals when the hot solution is slowly cooled.

151. *H_2SO_4:* white precipitate ($PbSO_4$): this precipitate is much less soluble in dilute H_2SO_4 than in water, hence H_2SO_4 should be added in excess to a pretty strong solution: pour off into two test-tubes and let the liquid stand; decant the liquid from the precipitate, and show that the precipitate may be dissolved by pouring upon it $H\bar{A}$, or better $H_2\bar{T}$ solution, then AmHO in excess and boiling; show also that it dissolves in boiling strong HCl.

152. *K_2CrO_4, or $K_2Cr_2O_7$:* yellow precipitate ($PbCrO_4$); pour off into two tubes, and show that the precipitate is soluble in KHO, but insoluble in $H\bar{A}$.

153. Mix some powdered $Pb\bar{A}_2.3H_2O$ with about twice as much Na_2CO_3 in a cavity on a piece of charcoal, and heat the mixture in the inner blowpipe flame; a yellow incrustation will form around the cavity and small lustrous globules of Pb will be seen within it. The incrustation is readily removed when heated in the inner blowpipe flame and colours the flame azure blue.

Detach one of the globules with the point of a knife, place it on the bottom of a mortar turned upside down, and give it a smart blow with the pestle; it flattens out without breaking into powder, showing that Pb is *malleable* and not *brittle.* If one of the globules be fixed upon the point of a penknife, it will be found by gentle friction on paper to mark it as a blacklead pencil does.

BISMUTH (Bi).—Use $Bi(NO_3)_3.5H_2O$ in dilute HCl.

154. *H_2S:* brownish black precipitate (Bi_2S_3):—

$$2Bi(NO_3)_3 + 3H_2S = Bi_2S_3 + 6HNO_3.$$

Decant most of the water; then pour some of the precipitate into three test-tubes, and show that it is insoluble in Am_2S and in KHO, but soluble in strong HNO_3.

155. *AmHO, or KHO:* white precipitate (BiH_3O_3); pour off a small quantity of the liquid and precipitate into a tube, add much AmHO and warm, the precipitate is undissolved; filter off the rest of the precipitate and keep it for the *following* reaction.

156. *H_2O:* pour *a few drops* of boiling dilute HCl upon the precipitate in the filter left from the last reaction, and allow the acid solution to drop from the funnel into a large quantity of distilled water: a *milkiness* appears, due to the formation of BiOCl: this precipitate may also be obtained by letting the original bismuth solution drop into much water:—$BiCl_3 + H_2O = BiOCl + 2HCl$.

Pour a little of the milky liquid into two test-tubes; add to one portion a little strong HCl and warm it, the precipitate dissolves; to the other portion add $H_2\bar{T}$ and warm, the milkiness remains, differing in this way from that caused by SbOCl.

If but little Bi is present, this milkiness often appears only after stirring and letting the liquid stand for five or ten minutes.

157. *K_2CrO_4, or $K_2Cr_2O_7$*, added after strong $Na\bar{A}$ solution gives a yellow flocculent precipitate of $Bi_2(CrO_4)_3$: add KHO, the precipitate does not dissolve, differing in this way from $PbCrO_4$; it dissolves, however, completely in HCl or HNO_3 added in excess.

The addition of $Na\bar{A}$ is necessary because bismuth chromate is soluble in HCl; the free HCl in the solutions is thus replaced by $H\bar{A}$, in which the precipitate is insoluble:—$Na\bar{A} + HCl = NaCl + H\bar{A}$.

158. *H_2SO_4:* no precipitate: difference from Pb.

159. Heat a mixture of powdered $Bi(NO_3)_3$ and Na_2CO_3 on charcoal in the inner blowpipe flame; an incrustation will be obtained which is *orange red* whilst hot and *yellow* when cold; also white globules of Bi will be seen, which are very brittle and easily crushed to powder by a sharp blow with the pestle.

COPPER (Cu).—Use $CuSO_4.5H_2O$.

Two classes of copper salts are known. *Cupric* salts are usually blue, but sometimes green; their colour commonly becomes very pale or disappears when the salts are thoroughly freed from water of crystallisation. *Cuprous* salts are usually white.

Cuprous oxide is red, and cupric oxide black.

160. *H_2S:* brownish black precipitate (CuS): filter, keeping the funnel carefully covered with a glass plate, as air quickly oxidises CuS to $CuSO_4$ and this dissolves and runs through with the filtrate. Place some of the precipitate in five test-tubes and show that it is insoluble in KHO, in Am_2S and in boiling dilute H_2SO_4, but dissolves in boiling HNO_3 and in KCy solution.

161. *AmHO,* if added in a diluted state and in very small quantity, gives a *greenish blue* precipitate; if more AmHO is added this precipitate dissolves, yielding an *intensely blue* liquid which contains *cuprammonium sulphate* $(N_2CuAm_4H_2)SO_4$; the solution becomes light blue again when an acid is added in excess.

162. H_2SO_4*:* no precipitate: difference from Pb.

163. K_4FeCy_6*:* reddish brown precipitate (Cu_2FeCy_6), insoluble in $H\bar{A}$; the colour is best seen when the reaction is tried in a white porcelain dish. In a *very dilute* solution only a reddish colour is seen.

164. *Fe:* if a bright piece of steel or iron, such as the blade of a penknife, which has been freed from grease by rubbing it with sand paper or by boiling it in a little dilute KHO, is dipped into $CuSO_4$ solution made acid with a few drops of H_2SO_4, it will be gradually covered with a red film of Cu.

165. *Zn and Pt:* if a piece of Zn be placed upon platinum foil or wire in $CuSO_4$ solution contained in a porcelain dish or watch glass, it causes a red film of Cu to deposit on the platinum: the $CuSO_4$ solution should be acidified with a few drops of H_2SO_4.

166. If finely powdered $CuSO_4.5H_2O$ and Na_2CO_3 are mixed in a cavity on charcoal and heated in the inner blowpipe flame, red scales of Cu are obtained; these are best seen after they have been washed (40).

167. *A borax bead* containing Cu is *green* while hot and *blue* when cold after having been fused in the outer blowpipe flame; fusion in the inner flame causes it to become *red* or *colourless* but only when very little Cu is present and the bead is long heated in a good reducing flame: the addition of a fragment of Zn or Sn aids this change.

168. *Flame coloration.*—If a loop of platinum wire is dipped into $CuSO_4$ solution and held in the inner blowpipe flame, or in the Bunsen flame, it gives a *green* coloration; the flame shows a *blue centre* after the loop has been moistened with HCl, or if $CuCl_2$ solution was originally used.

CADMIUM (Cd).—Use $CdSO_4.4H_2O$.

169. *H_2S:* bright yellow precipitate (CdS): boil off the H_2S and pour the liquid containing the precipitate into five tubes; show with these separate portions that the precipitate is insoluble in Am_2S, in KHO and in KCy solution, but soluble in boiling dilute HNO_3 and in boiling dilute H_2SO_4: dilute the H_2SO_4 solution with much water and pass H_2S for a short time, yellow CdS is reprecipitated.

170. *AmHO,* if added in a very diluted state and in small quantity, gives a white precipitate (CdH_2O_2); on adding more AmHO the precipitate readily dissolves.

171. *H_2SO_4:* no precipitate: difference from Pb.

172. If powdered $CdSO_4$ is mixed with Na_2CO_3 in a cavity on charcoal and heated in the inner blowpipe flame, it gives a brown incrustation.

173. GROUP II.A.—

Tests.	Hg—salts.	Pb—salts.
For liquids.		
1. Pass H_2S, or add H_2S water:	*Black* precipitate; when filtered and well washed, insoluble in boiling strong HNO_3, and *unchanged* by it.	*Black* precipitate; almost insoluble in boiling strong HNO_3, changed by it into *white* $PbSO_4$.
2. Add H_2SO_4:	——	White precipitate.
3. Add AmHO:	*White* precipitate, insoluble in excess.	*White* precipitate, insoluble in excess.
Blowpipe reactions for solids.		
4. Heated with Na_2CO_3 on charcoal in the inner blowpipe flame:	—— Test for Hg by (147).	*Yellow* incrustation; white *malleable* globules.

SEPARATION AND DETECTION OF METALS IN GROUP II.A.

175.* This separation depends upon the following differences:—

1. The solubility of Bi_2S_3, CuS and CdS in strong boiling HNO_3, in which HgS and PbS are insoluble and remain as HgS and $PbSO_4$ respectively.

2. The solubility of $PbSO_4$ in $H_2\bar{T}$ and excess of AmHO, in which HgS is insoluble.

3. The solubility of CuH_2O_2 and CdH_2O_2, and the insolubility of BiH_3O_3, in excess of AmHO.

4. The solubility of CdS and insolubility of CuS in boiling dilute H_2SO_4. The insolubility of CdS in KCy solution, in which CuS easily dissolves, furnishes another means of separation.

The presence of each metal is then confirmed by some special test.

TABLE OF DIFFERENCES.

Bi—salts.	Cu—salts.	Cd—salts.
Black precipitate; soluble in boiling strong HNO_3 to form a colourless solution.	*Black* precipitate; soluble in boiling concentrated HNO_3 to form a blue solution; insoluble in boiling dilute H_2SO_4.	*Yellow* precipitate; soluble in boiling HNO_3, and in boiling dilute H_2SO_4.
White precipitate, insoluble in excess. Confirm by (156).	*Blue* precipitate, easily soluble in excess to a deep blue liquid.	*White* precipitate, easily soluble in excess.
Orange red incrustation; white *brittle* globules.	No incrustation; red metallic scales. Confirm by (167, 168).	*Brown* incrustation; no globules.

176.* *A solution which may contain the metals of Group* II.A. is first made acid, if not already so, with a little HCl; whether the liquid is clear or not, H_2S is then passed in a rapid stream through it for about five minutes; the precipitate is filtered off; the clear filtrate is either mixed with H_2S-water, or it is diluted with a little water and H_2S is passed through it again for a short time, to ascertain whether the metals have been completely precipitated: if no further precipitate is produced, the filtrate may be rejected: but in case H_2S causes further precipitation, the gas must be passed for some time longer and the liquid poured again through the filter, the filtrate being rejected only when H_2S no longer produces any precipitate in it; the precipitate is then examined by Table II. (435), commencing at Group II.A. (Copper Group) and using only the left-hand side of the Table.

GROUP II.B.—ARSENIC GROUP.

177. This group includes As, Sb, Sn, Au, Pt.

Its members differ from those of Groups III.A., III.B., IV. and V. by being precipitated as sulphides by H_2S from a solution made acid with HCl; they differ from those of Group I. by not being precipitated by HCl.

The sulphides of this Group differ from those of Group II.A. by being soluble in Am_2S and in KHO.

178. Each metal of this group forms two series of compounds which resemble each other in many reactions, but also present certain differences. The two classes are distinguished conveniently by the terminations *-ic* and *-ous*, thus we speak of arsen*ic* and arseni*ous* acid.

ARSENIC (As).

Use for an arseni*ous* compound As_2O_3, dissolved in boiling water; and for an arsen*ic* compound $Na_2HAsO_4.12H_2O$ dissolved in water.

The tests described in paragraphs (179–186) give the same ultimate result with both arseni*ous* and arsen*ic* compounds: as a rule, however, the reaction proceeds slowly with arsen*ic* compounds, since its first stage consists in their conversion into arsenious compounds.

Arsen*ic* compounds are converted into arseni*ous* by boiling them with H_2SO_3, strong HCl, or other reducing agents; arseni*ous* compounds are oxidised into arsen*ic* compounds by being heated with HNO_3, or with other oxidising agents.

179. H_2S produces in neutral solutions of arsenious compounds only a yellow coloration; but if the solution is acidified with a few drops of HCl a light yellow flocculent precipitate (As_2S_3 forms immediately even in the cold.

Filter off some of the As_2S_3 and show that it is insoluble in boiling strong HCl. If some of the rest of the liquid containing the precipitate be poured into two test-tubes, the gradual addition of KHO or of Am_2S will completely dissolve the As_2S_3, which is reprecipitated, however, on the addition of HCl in excess.

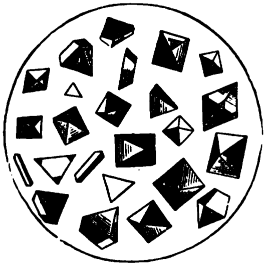
In cold acidified solutions of arsen*ic* compounds H_2S produces no precipitate; but if strong HCl is added and the solution is boiled, H_2S causes in the boiling solution first a precipitate of white S and then of yellow As_2S_3.

The complete precipitation of arsen*ic* solutions by H_2S is most rapidly effected by first adding H_2SO_3 to the boiling solution until it smells persistently of SO_2, then boiling until SO_2 is no longer smelt; the arsenious compound thus produced is readily and completely precipitated by H_2S in the cold solution.

180. *Reinsch's test.*—Several narrow strips of sheet copper are cleansed with sand paper, or by dipping them into strong HNO_3; they are then boiled in the arsenious solution, which has been previously acidified by dilute HCl: the surface of the copper will soon be coated with a dark grey film of (As_2Cu_5).

The strips are carefully removed, rinsed, gently pressed between filter paper, and then dried by heating them in the steam oven or by holding them between the finger and thumb in the flame: they are then gradually heated over a very small flame in a hot and dry test-tube; a sublimate of As_2O_3 will form, which will be crystalline and sparkling if moisture was entirely absent and the sublimation was effected very slowly while the sides of the tube were hot: these crystals if examined under the microscope are seen to be transparent, colourless, regular octahedra and tetrahedra (fig. 40), and their formation under the above conditions is a most valuable confirmation of the presence of As.

FIG. 40.



If the sublimate is white and non-crystalline it may be dissolved in a little boiling water, and the presence of As confirmed by applying tests (179, 187) to the solution.

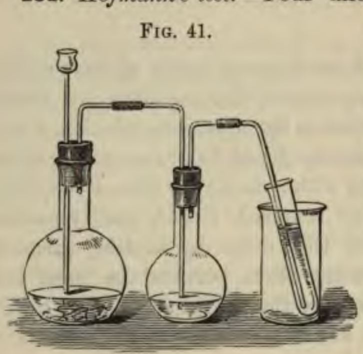
The presence of As in the grey deposit should always be confirmed by one of the above methods, since other metals may be deposited as grey films on the Cu.

This test serves to detect arsen*ic* compounds after their solutions have been reduced by being mixed with strong HCl or H_2SO_3 and boiled.

The following tests (181–184), which depend on the formation of *very poisonous* AsH_3 gas, should be performed in a cupboard provided with a good draught or in the open air.

181. *Hofmann's test.*—Pour dilute HCl upon some Zn in a small hydrogen flask fitted as is shown in (fig. 41). The hydrogen as it escapes bubbles through solution of lead acetate contained in the second flask, and is thus freed from any H_2S with which it might possibly have been mixed; it is then allowed to bubble through $AgNO_3$ solution contained in a test-tube: if the Zn and acid are free from As, as they should be, no precipitate or colour is produced by the gas in the $AgNO_3$ solution.

FIG. 41.



If *a little* As_2O_3 solution is now poured down the funnel into the hydrogen flask, a black precipitate of Ag will begin to form in the $AgNO_3$ solution; this is produced by the AsH_3 which is formed by the action of the nascent hydrogen upon the As_2O_3:—$As_2O_3 + 12H = 2AsH_3 + 3H_2O$.

The AsH_3 on passing into the $AgNO_3$ solution reacts thus:

$$AsH_3 + 6AgNO_3 + 3H_2O = 6Ag + H_3AsO_3 + 6HNO_3$$

Hence Ag is precipitated and H_3AsO_3 remains in solution.

As soon as a copious precipitate has been obtained, the test-tube is removed. It will be best to set aside the test-tube with its contents to be examined afterwards, and to proceed at once to use the gas, while it is being briskly evolved, for reactions (182, 183).

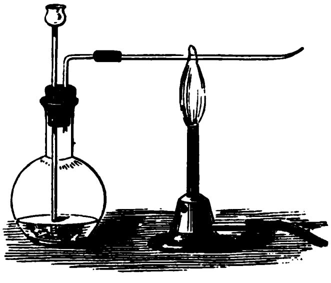
The liquid in the test-tube is filtered, the precipitate rejected, and several drops of $AgNO_3$ solution are added to the clear filtrate. Some very dilute AmHO is made by pouring several drops of AmHO solution into a test-tube, then nearly filling up with distilled water and inverting the tube. On mixing this drop by drop with the filtrate, a light yellow precipitate (Ag_3AsO_3 or Ag_2HAsO_3) will be obtained. The formation of this precipitate on the addition of AmHO is explained by the fact that H_3AsO_3 is in the solution in the presence of $AgNO_3$ and also of HNO_3, as is seen by the last equation; now $AgNO_3$ forms with H_3AsO_3 a yellow precipitate if no free acid is present in the liquid (187); hence on neutralising with AmHO the free HNO_3 which is already present, as well as that formed by the action of $AgNO_3$ on H_3AsO_3, the precipitate appears:—

$$H_3AsO_3 + 3AgNO_3 + 3AmHO = Ag_3AsO_3 + 3AmNO_3 + 3H_2O\,.$$

This precipitate is also readily soluble in AmHO; hence it is necessary to use *very dilute* AmHO and to add it gradually in order to avoid excess.

182. *Marsh's test.*—Disconnect the flasks and slip into the india-rubber joint a tube of hard glass about four inches in length, supporting it on a ring of a retort stand (fig. 42); then pour in through the funnel tube a little strong HCl.

FIG. 42.



If the experiment is performed as is here directed, the H will have been evolved for some time and will have expelled all air from the flask, therefore there will be no risk of an explosion occurring when the gas is lighted; but if the H is being produced for the first time, or the flask has been opened or recharged, allow the gas to escape briskly for four or five minutes; then ascertain that a test-tube full of it burns quietly before adding the liquid to be tested for As and lighting the gas at the jet. In any case it is well to cover the flask with a cloth before kindling the hydrogen (16).

On lighting the gas, its flame will be seen to differ from that of pure H by its livid blue colour and by giving off white fumes of As_2O_3. If these appearances are not noticeable, a little more As_2O_3 solution must be poured into the flask through the funnel tube.

Press down upon the flame *the inside* of a porcelain crucible lid or of a porcelain crucible or dish; a dusky black film of As will be deposited upon the cool surface; pour upon this some freshly made solution of $CaCl_2O$, the stain will be rapidly dissolved.

Also warm a stain of As inside a porcelain dish with Am_2S, it will dissolve and the solution will leave *yellow* As_2S_3 when it is slowly evaporated.

183. Add a little more strong HCl, if necessary, to cause the gas to come off rapidly; light it at the jet and heat a point near the middle of the tube strongly (fig. 42); a black mirror of As will form just beyond the part heated by the flame; the flame burning at the jet will meanwhile become colourless, showing that the arsenic has been removed from the hydrogen. Heat another point in the tube so as to produce a second mirror; then divide the tube between the mirrors, and dissolve one mirror when cold by warming it with some fresh solution of $CaCl_2O$ in a test-tube. Heat the other mirror gently in a test-tube held obliquely in the flame; a crystalline sublimate of As_2O_3 will be obtained (fig. 40). Dissolve the sublimate when cool in a little hot water and confirm the presence of As by reactions (179, 187).

183*a*. AsH_3 produces a characteristic colour with $HgCl_2$ solution. Remove the cork from the larger flask and push a plug of cotton wool into its neck to stop any spirting from the liquid in the flask; then cover the mouth of the flask with a small piece of filter paper moistened with $HgCl_2$ solution, the paper will become coloured first *yellow* then *brown*. If paper moistened with $AgNO_3$ solution be substituted it is *blackened*.

184. *Fleitmann's test.*—Boil some pieces of Zn in KHO solution, H will be evolved which is free from odour; if a little As_2O_3 solution is now added and the liquid is again boiled, a garlic odour will be perceptible owing to the evolution of AsH_3:—

$$\left\{\begin{array}{l} Zn + 2KHO = H_2 + ZnK_2O_2. \\ As_2O_3 + 12H = 2AsH_3 + 3H_2O. \end{array}\right.$$

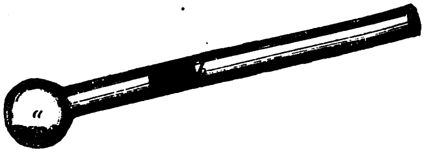
a small piece of filter paper moistened with $AgNO_3$ solution is placed on the mouth of the tube whilst the liquid is being boiled, it will be stained black by the separation of Ag (181).

This reaction does not succeed with arsenic compounds unless they have been reduced by being boiled with strong HCl or with H_2SO_3.

Sb compounds do not give this result, the test therefore distinguishes between As and Sb.

185. Mix any solid substance containing As, which has been finely powdered and thoroughly dried in the steam oven, intimately in a mortar with about four times as much of a mixture of KCy and freshly ignited Na_2CO_3; heat this mixture in a little bulb-tube of hard glass (fig. 43), or in a small test-tube about three inches in length, observing the precautions stated in the note under (147): a black mirror of As will form in the cooler part of the tube and frequently a smell of garlic may be detected at the mouth of the tube.

FIG. 43.



If the bulb is cut off and the mirror is gently heated in a test-tube, it will be converted into a white sublimate of As_2O_3 or into transparent octahedral crystals of this oxide (fig. 40).

186. If any solid substance containing As is mixed with Na_2CO_3 and the mixture is heated on charcoal in the inner blowpipe flame, a smell of garlic will be noticed when the charcoal is removed from the flame; also the flame will be coloured *livid blue*, unless this tint is overpowered by the yellow sodium coloration.

186*a*. If a little solid As_2O_3 is heated in a small ignition tube, it will be readily sublimed; if the As_2O_3 is perfectly dry, and is sublimed very slowly after the upper part of the tube has been first heated in the flame, the sublimate will sparkle and on examination under the microscope it will be seen to consist of octahedral crystals (fig. 40).

DISTINCTIVE TESTS FOR ARSEN*IOUS* AND ARSEN*IC* COMPOUNDS.

Reagent.	Arsen*ious* compound. Use As_2O_3 dissolved in H_2O.	Arsen*ic* compound. Use $Na_2HAsO_4.12H_2O$ dissolved in H_2O.
187. *$AgNO_3$*, several drops are added.	*Light yellow precipitate*, (Ag_2HAsO_3 or Ag_3AsO_3): this precipitate forms only on adding carefully, drop by drop, a little dilute AmHO :[*] show that it is dissolved by AmHO and by HNO_3. It is also appreciably soluble in $AmNO_3$: hence, if too much AmHO has been added, the precipitate does not always form on neutralising with HNO_3.	*Brown precipitate*, (Ag_3AsO_4): show that it is soluble in AmHO and in HNO_3.
188. *$CuSO_4$*, several drops:	*Yellowish green precipitate* ($Cu''HAsO_3$): this precipitate forms only on adding dilute AmHO carefully, drop by drop: show that it is soluble in AmHO and in HNO_3.	*Pale green precipitate* ($CuHAsO_4$): soluble in AmHO and in HNO_3.
189. *$CuSO_4$*, two drops: then KHO added:	The above yellowish green precipitate is first produced, but on addition of more KHO this dissolves to a clear *blue* liquid which on being boiled deposits *red* Cu_2O.	No clear blue solution is obtained, and no Cu_2O is produced on boiling the liquid.
190. *AmCl*, *AmHO*, and *$MgSO_4$*:	No precipitate.	White crystalline precipitate($MgAmAsO_4$): if filtered off and moistened with $AgNO_3$ solution on the filter it becomes *brown*.

---

[*] As_2O_3 when dissolved in water yields H_3AsO_3 which cannot give with $AgNO_3$ the Ag_3AsO_3 precipitate because this decomposition would liberate HNO_3 in which Ag_3AsO_3 is soluble ; hence the addition of AmHO is necessary to neutralise the free acid ; but since the precipitate is also soluble in AmHO, the latter must be added carefully in only just sufficient quantity to neutralise the acid. This is best effected by using some very dilute AmHO, made by pouring a few drops into a test-tube, filling the tube two-thirds with distilled water, then closing it with the thumb, and inverting it for a moment. The dilute AmHO is to be added drop by drop, shaking or stirring the test solution after each addition until the precipitate is obtained.

ANTIMONY (Sb).—Use $SbCl_3$ in dilute HCl.

Antimon*ic* and antimoni*ous* compounds give results ultimately identical in the following reactions (191–198) with the exception of (192). After these reactions, which are common to Sb in both its sets of compounds, two are given which are distinctive.

191. *H_2S:* orange red precipitate (Sb_2S_3): pour off into three tubes, and show that the precipitate is dissolved when it is warmed with pure KHO or with Am_2S, but is reprecipitated from these solutions on the addition of HCl in excess: also that it is soluble in boiling strong HCl.

192. If poured into much water, $SbCl_3$ solution yields a white precipitate or turbidity (SbOCl): this precipitate redissolves on adding HCl and warming and is also readily soluble in $H_2\bar{T}$; in this latter respect it differs from the similarly formed BiOCl precipitate (156).

193. If the acid $SbCl_3$ solution is poured upon some platinum foil in a porcelain dish and a piece of Zn is dropped upon the foil, the platinum will be rapidly coated with a black deposit of Sb. If the foil is rinsed with water and then boiled with HCl, the stain remains undissolved, but it is rapidly removed by boiling HNO_3.

Sb may be detected by the evolution of SbH_3 when a solution containing it is poured into a hydrogen apparatus as is directed in (181) for an arsenic solution; this gas differs from AsH_3 by being free from smell and from poisonous properties.

194. If SbH_3 is passed into $AgNO_3$ solution (181), it produces a black precipitate ($SbAg_3$):—

$$SbH_3 + 3AgNO_3 = SbAg_3 + 3HNO_3.$$

Filter off this precipitate and reject the filtrate, which will contain no Sb: wash the precipitate four or five times with boiling water upon the filter: then pour upon it boiling dilute $H_2\bar{T}$ solution, which will dissolve the Sb; receive the liquid in a test-tube; boil it, and pour it once more upon the filter; add HCl to the liquid, filter if necessary from AgCl and pass H_2S into the acid solution, orange red Sb_2S_3 will be precipitated.

195. A stain produced by Sb on porcelain in the manner already described in (182), differs from the As stain by presenting a *dead* surface and by being insoluble in $CaCl_2O$ solution; also when the stain is dissolved by warming it with Am_2S, the solution if evaporated by a gentle heat leaves *orange red* Sb_2S_3.

196. The Sb mirror, obtained as is described in (183), differs from the As mirror by being formed much nearer to the flame and on both sides of the heated part of the tube, owing to Sb being less volatile than As; it may furthur be distinguished by the test given in (195). This mirror, if oxidised by sublimation, is also insoluble in boiling water: if it is then dissolved in a little boiling HCl the solution gives an orange red precipitate (Sb_2S_3) when H_2S is passed into it, whereas the As_2O_3 solution gives yellow As_2S_3.

196*a*. SbH_3 acting on filter paper moistened with $HgCl_2$ solution, as is described in (183 *a*), yields a *greyish brown* stain.

197. If a solution containing Sb is boiled with Zn and KHO it does not evolve SbH_3, hence the escaping hydrogen does not stain paper moistened with $AgNO_3$ solution (184). This test distinguishes As from Sb.

198. If a little solid substance containing Sb is mixed in a cavity on wood charcoal with Na_2CO_3 and KCy and the mixture is heated in the inner blowpipe flame, a white incrustation forms on the charcoal and white globules of metallic Sb are obtained which are extremely brittle. The flame is seen to be coloured pale green, unless this tint is concealed by the yellow sodium coloration. When the melted Sb is withdrawn from the flame, each globule emits a white smoke and coats itself with sharp white crystals of Sb_2O_3.

The two distinctive tests (199, 200) for antimonious and antimonic compounds given in the following Table are seldom used; they may be read through without trying the reactions, and may be referred to hereafter if required for analytical purposes.

DISTINCTIVE TESTS FOR ANTIMON*IOUS* AND ANTIMON*IC* COMPOUNDS.

Reagent.	Antimon*ious*. Use the above solution of $SbCl_3$ in dilute HCl.	Antimon*ic*. Use $K_2Sb_2O_7$ solution, obtained by boiling some "Potassium metantimonate" with H_2O.
199. Add excess of KHO, then $AgNO_3$ solution:	A dark-coloured precipitate which when shaken after addition of AmHO leaves black Ag_4O undissolved.	A brown precipitate, which dissolves entirely on addition of AmHO.
200. Add excess of HCl and warm; then pour in a little KI solution:	No iodine is set free; proved by the liquid not turning brown and giving no colour after being cooled and mixed with freshly-prepared starch solution.	Iodine is liberated, giving a brown colour to the liquid and yielding a deep blue colour when starch solution is added to the cold liquid.

TIN (Sn).—Stannosum and Stannicum.

DISTINCTIVE REACTIONS FOR STANN*OUS* AND STANN*IC* COMPOUNDS.

Test.	Stann*ous*. Use $SnCl_2.2H_2O$ dissolved in dilute HCl.	Stann*ic*. Use $SnCl_4$ in dilute HCl.
201. Pass H_2S:	*Dark brown precipitate* * (SnS): soluble in KHO and in *yellow* Am_2S on heating; re-precipitated by HCl, from the KHO solution as brown SnS, from the Am_2S solution as yellow SnS_2. SnS is insoluble in *colourless* Am_2S.	*Yellow precipitate* * (SnS_2), often forming only when the liquid is heated: soluble in Am_2S (both *yellow* and *colourless*) and in KHO on heating, re-precipitated by HCl as yellow SnS_2 from both solutions.
202. $HgCl_2$:	*White precipitate* (Hg_2Cl_2): turns grey on being boiled if the $SnCl_2$ is in excess (145).	No precipitate.
203. $AuCl_3$: added after a drop of Br water:	*Purple* or *dark brown precipitate* of "Purple of Cassius."	No precipitate.

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* Both SnS and SnS_2 are readily soluble in boiling HCl.

The following reactions give ultimately the same results with both stann*ous* and stann*ic* compounds.

204. *Zn* when immersed in an Sn solution which has been acidified with HCl becomes coated with a spongy mass of Sn: the Zn should be allowed to remain for some time in the solution in a small porcelain dish; the action is much hastened by gentle heat. If the Zn rests on platinum foil the Sn is deposited in a spongy state *on the Zn, and does not stain the Pt black*, differing in this way from Sb. The deposition of Sn from $SnCl_2$ is much more rapid than from $SnCl_4$. Sn is often precipitated in this reaction in beautiful crystals.

The deposit of Sn may be rubbed and washed off the Zn in a porcelain dish, rinsed into a test-tube, and dissolved in a little boiling strong HCl; it will yield with $HgCl_2$ solution a white precipitate of Hg_2Cl_2 (202). This reaction depends upon the presence of $SnCl_2$ in the acid solution, and this is converted by exposure to the air into $SnCl_4$.

205. If any solid compound containing Sn be mixed with powdered Na_2CO_3 and KCy and heated on charcoal in the inner blowpipe flame, a slight white incrustation will be obtained, together with white particles of metallic Sn which are with difficulty fused into globules. The globules of Sn are malleable, but differ from those of Pb by their greater hardness, which prevents them from marking paper (153).

GOLD (Au).—Use $AuCl_3$ solution: note its light yellow colour.

$SnCl_2$ containing a little $SnCl_4$: purple or dark brown precipitate of *Purple of Cassius*, which is best seen when the test is made in a white porcelain dish.

$FeSO_4$ produces either at once, or on heating the solution, a very finely divided precipitate of Au: the liquid usually appears bluish by transmitted light and is always copper red and turbid by reflected light:—$2AuCl_3 + 6FeSO_4 = 2Au + 2Fe_2(SO_4)_3 + Fe_2Cl_6$.

H_2SO_3 produces on boiling a similar precipitate of Au: by boiling the liquid for some time in a porcelain dish the Au settles in small lack masses and the solution loses its colour.

PLATINUM (Pt).—Use $PtCl_4$ solution: note its reddish yellow colour.

AmCl added to a strong solution produces on standing for some time, or more quickly on being stirred, a yellow precipitate (Am_2PtCl_6), which is soluble in much hot water.

$SnCl_2$ gives a reddish brown colour ($PtCl_2$) in the solution acidified with HCl:—$PtCl_4 + SnCl_2 = PtCl_2 + SnCl_4$.

The method of separating and detecting Au and Pt is fully stated in paragraphs (472–474).

206. GROUP II.B.—TABLE OF DIFFERENCES.

The following tests answer for these metals in both sets of compounds: for further distinctive tests see the preceding reactions.

No. 3 may be omitted, unless decisive results have not been obtained by 1 and 2.

Tests.	As.	Sb.	Sn.
For liquids.			
1. Pass H_2S into the solution acidified with HCl and heat; if no precipitate forms heat to boiling, and again pass H_2S:	*Yellow* precipitate, insoluble in boiling strong HCl. Arsen*ic* compounds give this precipitate only on boiling: arseni*ous* in the cold.	*Orange red* precipitate, soluble in boiling strong HCl.	*Brown* precipitate with stann*ous*: *Yellow* precipitate with stann*ic*: Both soluble in boiling strong HCl.
2. Zn and HCl on platinum:	AsH_3 evolved, *no stain on the platinum.*	*Black stain of Sb on the platinum.*	Sn deposited on the zinc, *no stain on the platinum.*
3. Zn and HCl:	*AsH_3* gas is evolved, which if passed into $AgNO_3$ solution gives a black precipitate of Ag, H_3AsO_3 being left in solution; on adding *dilute* AmHO to this solution, yellow Ag_3AsO_3 is precipitated.	*SbH_3* gas is evolved, which if passed into $AgNO_3$ solution gives a precipitate of Ag_3Sb, from which hot $H_2\overline{T}$ solution dissolves Sb; H_2S gives in this solution after adding HCl orange-red Sb_2S_3.	*Sn* is deposited on the Zn strips: and if dissolved in boiling HCl gives a white precipitate on addition of $HgCl_2$.
For solids.			
4. Heat with Na_2CO_3 and KCy on charcoal in the inner blowpipe flame.	*No* metallic globules; smell of garlic. Confirm by heating same mixture in an ignition tube (185.)	*Brittle* metallic globules, white incrustation.	*Malleable* metallic globules, white incrustation.

SEPARATION AND DETECTION OF METALS IN GROUP II.B.

Several methods have been proposed for the separation and detection of As, Sb and Sn, when they are mixed; the most trustworthy are those introduced by *Hofmann* (208) and by *Fresenius* (210); but a method, which is more simple and can be much more rapidly carried out, is described in (209).

207.* *From a solution which may contain* As, Sb and Sn, these metals are first precipitated as sulphides by passing H_2S into the acidified solution; after filtering off the sulphides the filtrate is again tested by passing H_2S through it and warming, in order to see whether the precipitation has been complete. When no further precipitation is produced by H_2S, the precipitate is examined by (208, 209, or 210).

208.* *Hofmann's method* consists in pouring the solution of the metals into a flask, in which H is being generated by the action of HCl upon Zn; Sn will remain as a spongy mass on the Zn, while As and Sb will be evolved as AsH_3 and SbH_3; these two gases are passed into $AgNO_3$ solution and yield soluble H_3AsO_3 and insoluble Ag_3Sb, which are separated by filtration; after the three members of this group have been thus separated they are detected by the special tests already described, which are tabulated in (435 *b*).

209.* The following method of examining a precipitate, which may contain the sulphides of As, Sb, Sn (207), is simple and expeditious, and is sufficiently accurate when traces of these metals have not to be looked for.

It depends upon the insolubility of As_2S_3 in hot strong HCl, in which SnS, SnS_2 and Sb_2S_3 are soluble; the further separation of Sb and Sn is effected either by adding Zn alone to the acid solution, when Sb is evolved as SbH_3 and Sn is deposited upon the Zn; or by adding Zn and Pt, when Sn is deposited on the Zn and Sb on the Pt.

The precipitate produced by H_2S (207) is filtered off and allowed to drain for some time upon the filter in the funnel; it is best to drain it still further by carefully taking the filter out of the funnel, opening it out, and spreading it upon a piece of filter paper folded several times, or upon a pile of three or four cut filter papers. The precipitate is then removed to a small porcelain dish and heated for some time nearly to boiling with strong fuming HCl; the liquid is cooled and filtered :—

The residue will consist chiefly of As_2S_3, which is almost insoluble in strong HCl. Dry the washed residue on the filter at a gentle heat, then mix it with three or four times as much powdered KCy and Na_2CO_3, and heat the mixture in a small bulb-tube, removing any drops of water inside the tube by a twisted piece of filter-paper; a black mirror (185) :— *Presence of As.*	*The filtrate* may be examined for Sb and Sn by either I. or II. below :— I. Place a piece of platinum-foil in a porcelain dish and pour the acid filtrate upon it, then touch the foil with a piece of Zn; H will come off with effervescence, and if either at once or after a few minutes a black stain appears upon the platinum the *presence of Sb* is indicated.* The platinum-foil is removed and pieces of Zn are placed in the liquid : as soon as the bubbles of H cease to be given off, Zn still remaining undissolved, remove the pieces of Zn, rubbing and rinsing any dark deposit back into the dish; let this deposit settle, decant the liquid and heat the solid deposit with strong HCl for several minutes in a test-tube, dilute with a little water, filter if necessary, and add a few drops of $HgCl_2$ solution : a white or grey precipitate (204, 202) indicates the *presence of Sn.*
Note.—The As_2S_3 may also be dissolved by heating with a little fuming HNO_3. The excess of HNO_3 is boiled off and As detected as H_3AsO_4 by adding AmCl, excess of AmHO and $MgSO_4$ (190); or by addition of $AgNO_3$, and then cautiously neutralising with AmHO (187).	II. The acid filtrate is poured into a little hydrogen flask (fig. 41, p. 118), in which H has been coming off briskly for about five minutes, being produced by the action of a little strong HCl upon some pieces of Zn. The H is lighted at the jet, and the inside of a small porcelain dish or crucible lid pressed down upon the flame : a black stain not dissolved by solution of bleaching powder shows the *presence of Sb.* The residue in the flask is tested for Sn, as directed in the latter part of I. (above).

210.* *Fresenius' method* of detecting As, Sb and Sn, is very delicate and trustworthy. It depends upon the fact *that* fusion with Na_2CO_3 and $NaNO_3$ converts As_2S_3 into soluble Na_2HAsO_4, Sb_2S_3 into insoluble $Na_2H_2Sb_2O_7$, and SnS_2 into insoluble Sn or SnO_2.

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* The Sb thus deposited on the Pt may, after rinsing the foil, be dissolved by heating the Pt in a test-tube with a little very dilute HNO_3; on cooling, diluting, and passing H_2S, an orange-red precipitate will form, confirming the *presence of Sb.*

The precipitated sulphides (207) are dried on the filter, and the precipitate[*] well mixed on a watch glass or in an agate mortar with equal weights of powdered Na_2CO_3 and $NaNO_3$; this powder is dropped in small quantities at a time into some $NaNO_3$ kept melted in a small porcelain crucible over a Bunsen flame. When the powder has all been added, the crucible is heated sufficiently to keep the substance melted for a few minutes, and the fused mass is then poured into a small dry porcelain dish; as soon as the substance has cooled, cold water is poured upon it in the dish and also upon the residue adhering to the inside of the crucible, and the solid substance is allowed to soak for some time with occasional stirring, crushing it by pressure with a pestle if it does not easily fall to pieces; it is then filtered:—

The residue is washed on the filter with a mixture of equal measures of water and alcohol, to prevent solution of the $Na_2H_2Sb_2O_7$; the washings are rejected. The precipitate is then rinsed into a small porcelain dish, using as little water as possible; a few drops of strong HCl are added and the dish is heated, more HCl being added, if necessary, to make the liquid acid to litmus-paper. Whether the residue has dissolved or not, place in the liquid a piece of clean platinum-foil, and upon the foil a piece of pure Zn. Remove the foil after a few seconds, if it shows a black stain the *presence of Sb* is proved.[†] When the Zn ceases to be acted upon, Sn if present will remain as a spongy deposit. This is to be washed by decantation, dissolved by boiling with HCl in a test-tube, and $HgCl_2$ added immediately to the solution; a white precipitate (202) shows the *presence of Sn.*	*Filtrate:* HNO_3 is added until the solution is just acid after being boiled, then $AgNO_3$ solution is poured in as long as it causes any precipitate, and very dilute AmHO is added little by little[‡]; a brown precipitate, readily dissolving in excess of AmHO, shows the *presence of As.*

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[*] If the quantity of the precipitate is very small, the part of the paper containing the precipitate is cut up into small pieces and treated as the precipitate.

[†] See foot-note on page 130.

[‡] Instead of mixing dilute AmHO with the liquid, it may be carefully poured upon the top of the liquid; a brown colour is then seen at the surface of contact.

GROUP I.—SILVER GROUP.

211. This group includes Pb, Ag, Hg′.

The compounds of these metals differ from those of all other metals, by being precipitated as chlorides by HCl or by soluble chlorides; since, however, $PbCl_2$ is somewhat soluble in water, Pb is not entirely precipitated and is also found in Group II.A. as PbS.

LEAD (Pb).

The reactions for Pb have been already given under Group II.A. in paragraphs (149–153).

SILVER (Ag).—Use $AgNO_3$ solution.

212. *HCl:* white precipitate (AgCl), becomes curdy on being shaken or heated: pour off into four tubes; filter one, wash the precipitate and let it stand for some time exposed to sunlight or common daylight, it will become blackish purple; show with the other portions that AgCl dissolves readily when heated with AmHO or with KCy solution, and is precipitated again from these solutions on adding HNO_3 in excess; also that it is insoluble in HNO_3 even on boiling.

213. *H_2S or Am_2S:* black precipitate (Ag_2S), insoluble in Am_2S and in KHO, soluble in boiling dilute HNO_3.

214. *KHO:* brown precipitate (AgHO), insoluble in excess.

215. *AmHO:* light brown precipitate, produced only when *very dilute* AmHO is added drop by drop, since it is very easily soluble in excess of AmHO.

216. If any solid substance containing Ag is mixed with powdered Na_2CO_3 and heated on charcoal in the inner blowpipe flame, it will give white malleable globules or scales of Ag, but no incrustation.

MERCUROSUM (Hg').—Use $Hg'_2(NO_3)_2.H_2O$ in dilute HNO_3.

Reactions (144–148) for mercuric salts yield precisely similar results with mercurous salts; the two following reactions (217, 218), however, serve to distinguish mercuric from mercurous salts.

217. *HCl:* a white precipitate (Hg'_2Cl_2), insoluble in dilute acids unless warmed with both HCl and HNO_3, which convert it into soluble $Hg''Cl_2$: *becomes black when AmHO is poured upon it, but does not dissolve.* A solution of a mercur*ic* salt is not precipitated by HCl or by solutions of chlorides.

218. *AmHO, or KHO:* black precipitate, insoluble in excess. In a solution of a mercur*ic* salt AmHO usually causes a *white* precipitate (NH_2HgCl), and KHO precipitates yellow HgO.

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220. GROUP I.—TABLE OF DIFFERENCES.

Tests.	Pb.	Ag.	Hg′.
For liquids.			
1. Add HCl:	White precipitate, *soluble in boiling water; AmHO neither dissolves the precipitate nor changes its colour.*	White curdy precipitate, *insoluble in boiling water, easily soluble in warm AmHO.*	White precipitate, *insoluble in boiling water; insoluble in AmHO, but blackened by it.*
2. Add AmHO, very dilute:	*White precipitate,* insoluble in excess.	*Brown precipitate,* readily soluble in excess.	*Black precipitate,* insoluble in excess.
3. Add K_2CrO_4:	*Bright yellow* precipitate.	*Chocolate red* precipitate.	*Scarlet red* precipitate.
For solids.			
4. Fuse with Na_2CO_3 on charcoal in the inner blowpipe flame:	*White malleable globules of metal* which mark paper; yellow incrustation on the charcoal.	*White malleable globules or scales* which do not mark paper; no incrustation.	No metallic globules. Confirm by heating with Na_2CO_3 in an ignition tube (147).

SEPARATION AND DETECTION OF METALS IN GROUP I.

221.* The following differences are made use of:—

1. The solubility of $PbCl_2$ in boiling water, in which $AgCl$ and Hg'_2Cl_2 are insoluble.

2. The solubility of $AgCl$ in $AmHO$, in which Hg'_2Cl_2 is insoluble.

The presence of each member when separated is then confirmed by one of its special tests.

222.* *A solution which is to be examined for Pb, Ag, Hg'* is precipitated by adding HCl in excess, it is then filtered and the clear filtrate is tested with more HCl to ascertain that no further precipitate is produced: the precipitate is then examined by Table I. (433.)

The foregoing reactions only include those for the metals of common occurrence. For information concerning the reactions, detection and separation of the rarer metals and organic bases, the student is referred to the latter part of the sixth section of this book.

REACTIONS FOR ACID-RADICLES.

The acid-radicles are usually detected in the course of analysis by special tests: they cannot advantageously be precipitated in groups and the members of each group then separated and identified, as is the case with the metals. Accordingly the arrangement here adopted consists in placing together in a group such acid-radicles as somewhat resemble one another in their reactions, and at the end of each group stating upon what differences the detection of its members when occurring together depends.

The reactions for acid-radicles are worked through in the same way as those for the metals, a salt or other compound containing the radicle being employed. At the end of each group the student may with advantage detect one or more of its members, as was done with the metals.

The systematic Tables of Differences, such as are given for each group of the metals, are not drawn out for the acid-radicle groups; the student should have no difficulty in constructing such tables for himself, either mentally or on paper, by looking through the reactions.

GROUP I.—SULPHATE GROUP.

Sulphates are the only commonly occurring salts which give with $BaCl_2$ a precipitate insoluble in boiling HCl. Fluosilicates resemble sulphates in this respect, but they differ in so many other reactions that they are considered hereafter (299–301) in connection with fluorides and silicates, to which they are analytically more closely related. Selenates also yield a precipitate of $BaSeO_4$ on addition of $BaCl_2$, but it dissolves when boiled for some time with strong HCl.

SULPHATES ($-''SO_4$).—Use $Na_2SO_4.10H_2O$.

227. *$BaCl_2$, or $Ba(NO_3)_2$*: white precipitate ($BaSO_4$), insoluble when boiled with dilute HCl or HNO_3.

Add $BaCl_2$ or $Ba(NO_3)_2$ to strong HCl or HNO_3, a white precipitate will be produced consisting of $BaCl_2$ or $Ba(NO_3)_2$ which are insoluble in the strong acids: these precipitates are readily distinguished from $BaSO_4$ by their solubility in water.

228. If a sulphate, or any solid substance containing sulphur, is mixed with solid Na_2CO_3 and fused on charcoal in the inner blowpipe flame, it will yield Na_2S:—

$$Na_2SO_4 + 4C = Na_2S + 4CO.$$

The Na_2S is readily detected by detaching the cold solid mass from the charcoal with the point of a knife, placing a portion of it upon a bright silver coin, and moistening it with a drop of water: after the moistened mass has remained upon the coin for a short time it is rinsed off, and a black stain will then be seen upon the silver where the substance has rested (231).

Another portion of the cool mass may be moistened with a drop of HCl; a fetid smell of H_2S will then be observed, and a piece of paper moistened with $Pb\bar{A}_2$ solution and held above it, will be blackened (230).

This test is evidently only of value for detecting a sulphate when it is known that sulphur is not present in any other form in the substance to be tested. It is also necessary that sulphur should not be introduced in the Na_2CO_3, or by the flame: and since coal gas may contain sufficient sulphur to give the above reaction with pure Na_2CO_3, it is always advisable to employ the flame of a spirit lamp rather than that of a gas burner for this reaction.

Hydrogen sulphate, or sulphuric acid (H_2SO_4), may be detected by producing black charred stains on paper which has been dipped into it and dried by heat: it also has a strongly acid reaction to litmus, and evolves hydrogen when it is warmed with Zn: these properties are, however, shown *by* some metallic and acid sulphates.

GROUP II..—CARBONATE GROUP.

The five following classes of salts give off characteristic gases when acted upon by HCl.

CARBONATES (—$''CO_3$).—Use Na_2CO_3, or $CaCO_3$.

229. HCl, $H\bar{A}$, $H_2\bar{T}$, or almost any other acid, if poured upon a carbonate in a test-tube, causes colourless almost inodorous carbon dioxide gas (CO_2) to escape with effervescence; this gas is recognised by its property of turning lime or baryta water milky by the production in them of an insoluble carbonate.

The test may be tried in several ways.

1. The acid may be poured upon the carbonate in a test-tube: a glass rod which has been dipped into some perfectly clear lime water and has a small drop adhering to its end is then introduced into the test-tube, without touching the liquid or the inside of the tube; the drop will quickly become milky: the milkiness is best seen when the drop is held above a dead black surface.

2. Since CO_2 gas is much heavier than air, it will remain in the test-tube if the tube is held erect and its mouth is loosely closed with the thumb; by gradually sloping the tube the heavy gas may then be poured into another test-tube containing lime water, without allowing any liquid to flow out; on closing this tube with the thumb and shaking the lime water up with the gas, the liquid will become milky.

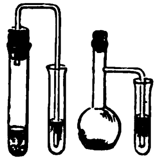
3. The acid may be poured upon the carbonate in a small beaker, which is immediately covered with a watch glass with its convex surface downwards and having a drop of clear lime water adhering to the middle: the drop will become milky.

4. If the CO_2 is given off in considerable quantity, it may be made to pass into some lime water contained in a separate tube; this is effected by either of the forms of apparatus shown in (fig. 44) on the next page.

In testing for a carbonate the following precautions must be attended to.

Since the milkiness produced in lime water by CO_2 gradually disappears when more CO_2 is absorbed, if the lime water does not become milky *at once* it must be constantly watched, in order to avoid the risk of its becoming milky and clear again before being seen.

FIG. 44.



Also if the addition of the first few drops of acid does not cause an effervescence of CO_2 more acid should be added, since many carbonates retain the first portions of CO_2 by forming acid carbonates, as is shown by the following equation:—

$$Na_2CO_3 + CO_2 + H_2O = 2NaHCO_3.$$

Hydric carbonates, or bicarbonates, are decomposed by heat into normal carbonates with escape of CO_2 and H_2O: prove this by adding $NaHCO_3$ to boiling water in a test-tube, when effervescence will occur:—

$$2NaHCO_3 = Na_2CO_3 + CO_2 + H_2O.$$

A solid substance which is being tested for a carbonate by HCl should be first moistened so as to drive out the air bubbles entangled in it, since these whilst escaping might be mistaken for a slight effervescence of CO_2.

A solution of a bicarbonate made in *cold* water gives no precipitate with $MgSO_4$ solution, but after being boiled the solution will contain a normal carbonate and will precipitate white $MgCO_3$.

With $HgCl_2$ the solution of a bicarbonate yields a *pale yellow* precipitate, while solution of a normal carbonate yields a *red* precipitate.

Hydrogen carbonate, or carbonic acid (H_2CO_3), can only exist in dilute aqueous solution; addition of lime water to its solution causes milkiness; but since other substances in solution behave in a similar way, the acid is best found by boiling the liquid and passing the CO_2 which is evolved with the steam into lime water; the lime water becomes milky.

SULPHIDES ($-''S$).—Use FeS or Am_2S.

230. *HCl or H_2SO_4* when poured upon a small fragment of FeS causes H_2S gas to escape with effervescence: this gas is detected by its fetid smell, resembling that of rotten eggs, and by its property of blackening $Pb\bar{A}_2$ solution, owing to the formation of PbS. The gas may be made to act upon the $Pb\bar{A}_2$ solution by the methods given for CO_2 (229. 1–4);

or a small strip of filter paper, moistened with the solution, may be introduced into the gas on the end of a glass rod.

This test is made much more delicate if the paper is moistened with a solution prepared by adding KHO to boiling $Pb\bar{A}_2$ solution until the precipitate formed at first is redissolved: a test paper thus prepared becomes intensely blackened by H_2S. The paper strip should be attached to the rod by only half its length, so as to expose both sides of the free end to the gas.

231. A solution of a sulphide if dropped upon a bright silver coin produces a black stain (Ag_2S); this may be removed by rubbing the coin with moist lime.

232. An insoluble sulphide such as FeS, which gives off H_2S on the addition of HCl, produces a black stain when it is placed on a silver coin and moistened with a drop of HCl.

233. *$Pb\bar{A}_2$* solution gives with soluble sulphides a black precipitate (PbS). The solution produced by adding KHO in excess to $Pb\bar{A}_2$ solution is a more delicate reagent for this test.

234. Free S and many sulphides, when strongly heated in a test-tube, give a sulphur sublimate of brown drops or yellow powder: and if heated in a tube open at both ends and held obliquely in the flame they give off SO_2 gas, which may be recognised by its pungent smell and by turning paper moistened with $K_2Cr_2O_7$ solution green.

235. Reaction (306) for a cyanide may be employed as a very delicate test for a soluble sulphide.

An insoluble sulphide, when fused in a covered porcelain crucible or in a closed hard glass tube with Na_2CO_3, yields soluble Na_2S.

A sulphide when fused with KNO_3 or when heated with strong HNO_3 yields a sulphate, which may be detected in solution by $BaCl_2$ (227).

Hydrogen sulphide, or hydrosulphuric acid (H_2S), in aqueous solution is easily recognised by (231) or (233); unless the solution is very dilute it emits H_2S, which is readily detected by its smell, or by suspending lead acetate paper over the liquid.

SULPHITES ($-''SO_3$).—Use Na_2SO_3.

236. *HCl or H_2SO_4* when poured upon Na_2SO_3 and warmed gives off SO_2 gas, which is known by its pungent smell and by turning $K_2Cr_2O_7$ solution green: a drop of the $K_2Cr_2O_7$ solution hanging on the end of a glass rod may be exposed to the gas (229), or a small strip of filter paper may be wetted with the bichromate solution and then attached by half its length to the rod, the other half hanging freely in the gas. $H_2\bar{T}$ solution does not evolve SO_2 from a sulphite.

237. *$BaCl_2$:* white precipitate ($BaSO_3$): entirely soluble in HCl, unless Na_2SO_4 is also present when $BaSO_4$ remains undissolved: on adding Cl- or Br-water to the HCl solution, $BaSO_4$ is formed and precipitated:—

$$BaSO_3 + 2HCl = BaCl_2 + H_2SO_3.$$

$$BaCl_2 + H_2SO_3 + H_2O + 2Cl = BaSO_4 + 4HCl.$$

238. The addition of *Na_2SO_3* solution to a mixture of *HCl and Zn*, which is giving off pure H, immediately causes an evolution of H_2S: the H_2S is detected by its smell, or by blackening a piece of filter paper moistened with solution of $Pb\bar{A}_2$ or of PbO in excess of KHO (230).

Hydrogen sulphite, or sulphurous acid (H_2SO_3), can be detected in aqueous solution by adding a drop of $K_2Cr_2O_7$ solution, which will at once become green: strong aqueous solution slowly evolves SO_2, which is detected by its smell; dilute solutions also evolve SO_2 when they are heated.

THIOSULPHATES, OR HYPOSULPHITES ($-''S_2O_3$).

Use $Na_2S_2O_3.5H_2O$ solution.

239. *HCl or H_2SO_4* causes no immediate change in the *cold* solution, but the acidified liquid after standing for a few seconds deposits a precipitate of yellow S and then contains H_2SO_3; hence SO_2 may be recognised by its pungent smell when the liquid is heated and may be detected in the solution by adding a few drops of red $K_2Cr_2O_7$ solution, which changes colour to green.

240. *Fe_2Cl_6* gives a reddish violet colour, which vanishes after a short time in the cold and immediately when heated; the Fe_2Cl_6 solution at the same time loses its yellow colour owing to the change of Fe_2Cl_6 into $FeCl_2$.

240*a*. *$AgNO_3$;* a white precipitate ($Ag_2S_2O_3$), becoming black after standing for a short time, or immediately when heated, owing to the formation of Ag_2S:—

$$Ag_2S_2O_3 + H_2O = Ag_2S + H_2SO_4.$$

The above precipitate ($Ag_2S_2O_3$) dissolves very readily in excess of the $Na_2S_2O_3$ solution, hence it is most easily obtained by dropping the latter into some $AgNO_3$ solution.

Hydrogen thiosulphate, or thiosulphuric acid ($H_2S_2O_3$), is extremely unstable, rapidly separating into $S + H_2SO_3$ (239).

HYPOCHLORITES ($-'ClO$).—Use $CaCl_2O$.

Hypochlorites in the solid state or in strong solution give off a faint smell of Cl.

241. *HCl, H_2SO_4, $H_2\bar{O}$, or even CO_2* sets free Cl from a hypochlorite in the cold; the gas may be recognised by its smell, its yellowish colour, and by bleaching a piece of moistened litmus paper which is held for a short time in the tube: if the Cl is small in quantity and remains dissolved, a piece of litmus paper may be shaken up with the solution.

Bleaching by the solution may occur even without the addition of acid, owing to the bleaching power of the hypochlorite itself or to the liberation of Cl by the CO_2 in the air: on the addition of any acid, however, the bleaching of the immersed paper is very rapid:—

$$CaCl_2O + 2HCl = 2Cl + CaCl_2 + H_2O.$$

242. If a solution of a hypochlorite is added in large quantity to $Pb\bar{A}_2$ solution it gives a white precipitate which becomes red and then dark brown (PbO_2) when the liquid is boiled for a short time.

When a hypochlorite is heated with $MnCl_2$ solution, it also gives a *dark brown* precipitate.

Hydrogen hypochlorite, or hypochlorous acid ($HClO$), is a yellow liquid with sweetish smell, and is very unstable unless largely diluted: it bleaches litmus and evolves Cl when it is warmed with HCl.

NITRITES ($-'NO_2$).—Use KNO_2.

243. When a nitrite is warmed with dilute H_2SO_4 it evolves reddish brown *nitrous fumes*, which have a characteristic smell; they are best seen by looking down the tube at a white surface.

244. When cold $FeSO_4$ solution is added to the solution of a nitrite it becomes brown; the colour becomes much more intense on the addition of cold dilute H_2SO_4: it is due to a compound of NO with $FeSO_4$, and is destroyed by boiling the liquid, when red fumes are given off (243).

245. Drop *a little* starch powder into some water boiling in a porcelain dish and boil for a short time; then cool the starch solution and add to it a few drops of KI solution, then some solution of a nitrite and several drops of $H\bar{A}$: an intense blue colour will be produced, owing to the combination of iodine, set free by the HNO_2, with the starch; the test is best made in a white porcelain dish, the liquid, which often appears black at first, being diluted if necessary until its blue colour becomes visible. This is a most delicate test if the liquid is quite cold: and it is of value if the colour is not produced until the $H\bar{A}$ is added.

Hydrogen nitrite, or nitrous acid (HNO_2), is extremely unstable, decomposing rapidly into HNO_3, NO, and H_2O.

DETECTION OF ACID-RADICLES IN GROUP II.

246.* *The detection of these acid-radicles* when occurring singly presents no difficulty; but most of the acids which are formed on the addition of HCl to the salts of this group, react upon one another; thus HClO oxidises HNO_2, H_2S and H_2SO_3; and H_2SO_3 oxidises H_2S. The analyst may often by skilful use of the above reactions detect these acid-radicles when they are mixed; but their detection in certain mixtures is a problem only soluble by careful consideration and not unfrequently insoluble.

SO_2 may be detected by $K_2Cr_2O_7$ paper: H_2S by $Pb\bar{A}_2$ paper: Cl by bleaching moist litmus paper: N_2O_3 by its brown colour. But CO_2 can only be detected in the presence of much SO_2 by passing the gases through hot $K_2Cr_2O_7$ solution which absorbs SO_2, CO_2 passing on and being detected by lime water.

It should be noted that while carbonates evolve CO_2 when acted upon by $H_2\bar{T}$, sulphites are not decomposed by that acid.

GROUP III.—NITRATE GROUP.

This group includes nitrates and chlorates: these salts resemble one another in many respects, more particularly in the fact that none of them are perfectly insoluble in water; hence no method of precipitating them is known.

NITRATES ($-'NO_3$).—Use KNO_3.

247. Add to some KNO_3 solution contained in a rather broad test-tube, about an equal bulk of strong H_2SO_4; cool the liquid in a stream of cold water from the tap; then pour some strong cold $FeSO_4$ solution in a gentle stream from a pipette down the inside of the tube, which is held in an inclined position: a distinct layer of the $FeSO_4$ solution may thus be formed on the surface of the acid liquid. A *dark brown* film or ring, which sometimes extends upwards, will then appear where the liquids are in contact.

The colour is most easily seen by holding a piece of filter paper between the tube and a bright flame or window, and looking through the tube at the light. A mere *yellow* colour is often produced in the absence of a nitrate and may be disregarded.

The test is made more delicate by letting the tube stand in a beaker of cold water for a few minutes, since heat prevents or removes the brown coloration. If much nitric acid is present, the brown colour extends upwards on mixing the acid and $FeSO_4$ solution, until the heat evolved by the dilution of the strong H_2SO_4 causes the NO to escape and to form brown fumes in the test-tube; the liquid at the same time loses its dark brown colour (244).

248. Put a few small scraps of Cu in some KNO_3 solution and add strong H_2SO_4; brown nitrous fumes will appear either at once or on warming the tube: they are best seen, if small in quantity, by looking down the tube at a white surface.

The liquid at the same time becomes blue from the production in it of Cu $(NO_3)_2$:—

$$8HNO_3 + 3Cu = \overline{2NO} + 3Cu(NO_3)_2 + 4H_2O.$$

NO is itself a colourless gas, but it yields N_2O_3 and N_2O_4 by oxidation when mixed with air, and these are reddish brown.

249. If some cold dilute indigo solution is poured into cold KNO_3 solution until it is decidedly blue and then H_2SO_3 is added, the blue colour remains unchanged: this reaction distinguishes a nitrate from a chlorate.

250. If any solid nitrate is heated to redness with fusion mixture $(Na_2CO_3 + K_2CO_3)$ on platinum foil for several minutes, a soluble alkaline nitrite is formed; if the foil after cooling is boiled in water, a portion of this solution may be proved to contain a nitrite by the test in paragraph (245); another portion acidified with HNO_3 will be found to give no precipitate with $AgNO_3$. Compare (254).

A nitrate in solution may also be reduced to a nitrite by the action of nascent hydrogen. Add dilute H_2SO_4 to some KNO_3 solution; on the addition of freshly made starch solution containing KI no colour will be produced, but on dropping in a fragment of Zn the liquid will assume a deep blue colour (245).

251. Solid KNO_3 when fused on charcoal in the blowpipe flame *deflagrates*, that is to say the surface of the charcoal burns away rapidly like gunpowder.

251*a*. Many metallic nitrates, when heated in a glass tube closed at one end, evolve reddish brown fumes with a characteristic smell (N_2O_4) and oxygen: $PbN_2O_6 = N_2O_4 + O + PbO$. The N_2O_4 is recognised by its colour and smell, and the O by inflaming a glowing splinter of wood. Lead nitrate may be used in trying this experiment.

Hydrogen nitrate, or nitric acid (HNO_3), has a strongly acid reaction: it gives with $FeSO_4$ solution a brown ring

(247), and with Cu brown fumes (248) and a blue solution. When it is evaporated with quill clippings, it stains them bright yellow.

CHLORATES ($-'ClO_3$).—Use $KClO_3$.

252. If cold dilute indigo solution is gradually added to a *cold* solution of $KClO_3$ until the liquid is coloured faintly but distinctly blue, and some H_2SO_3 is then poured in and the mixture is shaken, the blue colour of the indigo is at once destroyed (249).

253. *Strong H_2SO_4* poured upon *a little* solid $KClO_3$ becomes orange red in colour, and evolves when shaken a bright yellow gas (Cl_2O_4):—

$$3KClO_3 + H_2SO_4 = \overline{Cl_2O_4} + KClO_4 + K_2SO_4 + H_2O.$$

This gas has a smell somewhat resembling that of chlorine; it explodes readily when gently heated, hence on warming the mixture in the tube crackling sounds or small explosions will be produced.

This experiment may be dangerous, unless very little $KClO_3$ is used and the mixture is heated gently with the mouth of the test-tube turned away from the person.

254. *$AgNO_3$* produces no precipitate with $KClO_3$ solution which is free from KCl. But if some solid $KClO_3$ be heated in a test-tube, it fuses and gives off oxygen with effervescence; this gas may be recognised by holding in the tube a slip of wood with a spark at its end, the spark is caused to burst into a flame.

If the tube is then allowed to cool and the residue is dissolved by heating it with water, a portion of the solution will be found to give no reaction for a nitrite (245): but another portion will give on addition of $AgNO_3$ a white precipitate (AgCl), which does not dissolve in HNO_3 even on boiling, but dissolves readily in AmHO (257): this precipitate is caused by KCl formed from the chlorate: $KClO_3 = KCl + O_3$.

A chlorate which does not yield a soluble chloride must be mixed with fusion mixture before it is heated.

This reaction readily distinguishes a chlorate from a nitrate.

255. If $KClO_3$ is heated on charcoal in the blowpipe flame, it causes the charcoal to deflagrate.

Hydrogen chlorate, or chloric acid ($HClO_3$), is a colourless, odourless liquid, which first reddens and then bleaches litmus paper; on being kept for some time it changes into O, Cl, $HClO_4$ and H_2O; the same change occurs rapidly when it is heated.

DETECTION OF A NITRATE AND A CHLORATE.

256.* There is little difficulty in distinguishing a nitrate from a chlorate.

When present together they more or less interfere with one another's reactions; but in the absence or after the removal of chlorides and nitrites, they may be detected by heating the solid substance strongly for a short time, and then testing for a nitrite and a chloride (250, 254).

GROUP IV.—CHLORIDE GROUP.

Chlorides, bromides and iodides closely resemble one another in their reactions; they are all precipitated by $AgNO_3$ from solutions to which HNO_3 has been added in excess; in this respect they differ from all salts except cyanides, ferrocyanides and ferricyanides and these are easily distinguished by other means.

CHLORIDES (– 'Cl).—Use NaCl.

257. *$AgNO_3$* when added to the solution of a chloride gives a *pure white* precipitate (AgCl), which collects into curdy masses if the liquid is heated or shaken, and rapidly turns violet in sunlight, slowly darkening even in diffused daylight.

Decant the water; warm one portion of the precipitate with HNO_3, it will not dissolve; to another portion add AmHO, it will readily dissolve; AgCl is also soluble in KCy and in $Na_2S_2O_3$ solutions. Decant the liquid and warm the precipitate with a little strong H_2SO_4, the acid will remain colourless and no coloured vapour will be given off.

258. Warm some solid NaCl with strong H_2SO_4, colourless HCl will be given off and may be recognised by fuming in the air, by reddening moistened blue litmus paper, and by making a drop of $AgNO_3$ solution acidified with HNO_3 milky (229. 1–4).

259. Mix together intimately some finely powdered NaCl and MnO_2, then add strong H_2SO_4 and warm the mixture; chlorine gas will be evolved:—

$$2NaCl + MnO_2 + 3H_2SO_4 = 2Cl + MnSO_4 + 2NaHSO_4 + 2H_2O.$$

This gas is recognised by its power of bleaching a piece of moistened litmus paper, which is introduced into the tube on a glass rod. The most delicate way of making this test is to mix the substances in a small beaker, cover this with a watch glass which bears on its under surface a piece of moist litmus paper, and then warm the mixture and let it stand for some time. Moist starch is not coloured when held in the escaping gas.

Many samples of commercial MnO_2 evolve Cl when heated with H_2SO_4; hence unless the MnO_2 has been specially prepared by precipitation, it must be carefully tested before it is used for this reaction; or the MnO_2 and H_2SO_4 may be first boiled together as long as any bleaching action is produced when moist litmus is held in the tube, then the substance to be tested is added and Cl is again tested for while the mixture is being heated.

260. If an intimate mixture is made of solid NaCl with three or four times as much $K_2Cr_2O_7$ by rubbing the two substances together to a fine powder in a mortar, and this mixture is stirred with strong H_2SO_4 and heated in the tube or flask represented on page 138, reddish brown vapour (CrO_2Cl_2) will be evolved:—

$$4NaCl + K_2Cr_2O_7 + 6H_2SO_4 = 2CrO_2Cl_2 + 4NaHSO_4 + 2KHSO_4 + 3H_2O.$$

If the vapour escaping from the delivery-tube is passed into some water in a test-tube, the water will become reddish yellow in colour, owing to the formation of H_2CrO_4:—

$$CrO_2Cl_2 + 2H_2O = H_2CrO_4 + 2HCl.$$

On adding excess of AmHO to this reddish yellow liquid the colour changes to pale yellow, since Am_2CrO_4 is formed. On now adding excess of $H\bar{A}$ the original reddish yellow colour is reproduced, and the presence in this liquid of H_2CrO_4, and therefore indirectly that of a chloride, may be shown by the formation of a yellow precipitate ($PbCrO_4$) on the addition of $Pb\bar{A}_2$ solution.

The detection of a chloride by this method depends on the formation of H_2CrO_4 in the water in the test-tube from the vapour of CrO_2Cl_2: great care must therefore be taken that none of the mixture containing a chromate is allowed to get into the delivery-tube and thence into the water.

Care must also be taken that the water is not sucked back into the hot strong H_2SO_4: this is prevented by raising the delivery-tube out of the water as soon as the heating is stopped.

Hydrogen chloride, or hydrochloric acid (HCl), is a colourless gas which fumes in the air and dissolves very readily in water; both the gas and its solution render milky some $AgNO_3$ solution which has been acidified with HNO_3. When it is heated with MnO_2 the acid evolves chlorine gas.

BROMIDES ($-'Br$).—Use KBr.

261. *$AgNO_3$* when added to the solution of a bromide gives a *yellowish white* precipitate ($AgBr$), which is easily coagulated by heating or shaking the liquid; it is insoluble in HNO_3, easily soluble in KCy and in $Na_2S_2O_3$ solutions, and slightly soluble in AmHO; it therefore resembles $AgCl$, but is distinguished from it by its colour. If the liquid is decanted and the precipitate is heated with strong H_2SO_4, no violet vapour will be evolved as is the case with AgI.

262. Solid KBr when heated with strong H_2SO_4 gives off acid fumes (HBr) and *reddish brown* vapour of Br; this vapour somewhat resembles Cl in smell and in bleaching power, but differs from it in colour. Bromine vapour also has the power of staining cold moist starch *orange red:* the starch powder may be taken up on the wetted end of a glass rod, and moistened by breathing upon it several times. If MnO_2 is mixed with the KBr powder before adding H_2SO_4, the Br is evolved in greater quantity and more readily (259).

263. *Chlorine water*, or $CaCl_2O$ solution acidified with HCl, if added carefully drop by drop to the solution of a bromide, liberates Br, which colours the solution orange red; excess of Cl must be carefully avoided, since it combines with the Br and destroys its colour: $KBr + Cl = Br + KCl$.

On warming a part of this solution, reddish brown Br vapour is given off, which stains cold moist starch *orange red* (262).

If CS_2 is added to another portion of the solution of Br and the liquids are well shaken together, the Br will be dissolved away from the water by the CS_2, and on standing the reddish brown CS_2 solution will sink beneath the colourless water; on adding a little KHO solution and again shaking the liquids up together the colour of the Br will disappear from the CS_2; this is due to the formation of the colourless salts KBr and $KBrO_3$, which remain in solution in the water:—

$$6Br + 6KHO = 5KBr + KBrO_3 + 3H_2O$$

264. If an intimate mixture of solid KBr and $K_2Cr_2O_7$ is heated with strong H_2SO_4 (260), it will evolve reddish brown bromine vapour:—

$$6KBr + K_2Cr_2O_7 + 11H_2SO_4 = 3Br_2 + Cr_2(SO_4)_3 + 8KHSO_4 + 7H_2O.$$

If the Br vapour is passed into water it will colour it reddish brown; CS_2, if shaken up with this brown solution, will dissolve away the Br and settle beneath the colourless water as a brown layer; on shaking the brown solution in water or in CS_2 with AmHO or KHO, the colour of the solution will be destroyed. This reaction serves to detect a chloride in the presence of a bromide.

Hydrogen bromide, or hydrobromic acid (HBr), is a gas resembling HCl; its solution differs by evolving Br vapour when it is heated with MnO_2.

IODIDES (– 'I).—Use KI.

265. *$AgNO_3$* when added to the solution of an iodide, gives a *yellow* precipitate (AgI), which is easily coagulated by heating or by shaking the liquid: it is insoluble in HNO_3 and very slightly soluble in AmHO, but is easily soluble in KCy and in $Na_2S_2O_3$ solutions.

This precipitate differs from AgCl and AgBr by its colour and by its slight solubility in AmHO, but more particularly by its behaviour when heated with strong H_2SO_4. If the liquid is decanted from the precipitate, and the precipitate is then heated with strong H_2SO_4, the acid becomes coloured

red, and violet vapour of iodine is seen on looking down the tube; the colour is most distinctly seen after the tube has been allowed to cool for a short time. Even traces of iodine vapour may be detected by holding in the tube a glass rod which has been dipped into freshly made starch solution, the starch will be coloured blue.

The starch solution is made by throwing *a little* starch powder into water boiling in a small porcelain dish, boiling for a few seconds and then cooling the solution.

266. *Cu_2SO_4*, obtained by mixing $CuSO_4$ solution with H_2SO_3 or with $FeSO_4$ solution, gives in the solution of an iodide a white precipitate (Cu_2I_2); gentle warming, or partially neutralising the free acid by AmHO, will cause this precipitate to form more quickly:—

$$2CuSO_4 + H_2SO_3 + H_2O = Cu_2SO_4 + 2H_2SO_4$$
$$2KI + Cu_2SO_4 = Cu_2I_2 + K_2SO_4 .$$

If this precipitate is separated by decantation or filtration, and is then warmed with strong H_2SO_4, the acid becomes red and violet iodine vapour is evolved.

Solutions of chlorides and bromides are not precipitated by Cu_2SO_4.

267. If a solid iodide is warmed with strong H_2SO_4, acid fumes (HI) and violet iodine vapour will be evolved; if the iodine vapour is large in quantity, it will condense on the inside of the tube as a black solid; this vapour colours starch solution blue, as may be proved by holding in it a rod or strip of paper moistened with freshly made starch solution, or by pouring the heavy fumes out into a white porcelain dish, the inside of which has been wetted with the starch solution. Iodine is more readily and plentifully evolved if MnO_2 is mixed with the iodide before adding H_2SO_4 (259).

268. *Chlorine water*, or $CaCl_2O$ solution acidified with HCl, added carefully drop by drop to the solution of an iodide sets iodine free, which dissolves in the water giving a brown liquid; addition of Cl in excess must be carefully avoided, as it would form ICl_3, which is colourless and does not give the following reactions. Divide this brown liquid into three parts.

Heat one part; violet iodine vapour is given off and is best seen by looking down the mouth of the tube at a white surface. The colour is most distinctly seen if some strong H_2SO_4 is added to the liquid before heating it. If a glass rod is dipped into starch solution and held in the vapour, the starch becomes blue.

To another portion add starch solution, an intense blue colour is produced which often appears black unless much starch solution is added and the liquid is largely diluted; on warming the solution it becomes colourless, but it often regains its colour on being cooled. The solution must be diluted and the starch added in excess, to insure the removal of the colour by heat.

To the third portion add a drop of CS_2 and shake well; the CS_2 dissolves the iodine and gradually forms a beautiful violet layer beneath the water. Add a little KHO solution and shake the liquids together, the violet colour of the CS_2 will disappear owing to the formation of the colourless salts KI and KIO_3.

269. If a little KNO_2 is dissolved in the solution of an iodide and any dilute acid is added, HNO_2 is produced and liberates the iodine. A solution of N_2O_4 in dilute H_2SO_4 also sets free iodine from Kl (546. 4). The iodine thus liberated in solution may be identified by the three methods described in (268), of which the starch and the CS_2 tests are the most delicate.

These two reagents for liberating iodine have the advantage over Cl that they do not hinder its detection if they are added in excess; they are also useful because they do not liberate Br from its compounds.

270. *$HgCl_2$* solution gives with the solution of an iodide a scarlet precipitate (HgI_2), which is easily soluble in excess of $HgCl_2$ or of the iodide; with a solution of a bromide or chloride $HgCl_2$ gives no precipitate.

$Pb\bar{A}_2$ gives with the solution of an iodide a *bright yellow*

precipitate (PbI_2); with the solution of a bromide or a chloride it gives a *white* precipitate.

Hydrogen iodide, or hydriodic acid (HI), is a gas resembling HCl and HBr; its solution differs by evolving iodine when it is heated with MnO_2.

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271. *A chloride, bromide, or iodide,* is easily detected by the foregoing tests. The reactions given in paragraphs (259) and (260) are perhaps the most characteristic tests for a chloride, those described in (262), (263) and (264) for a bromide, whilst an iodide is detected with the greatest ease and certainty by reaction (269).

The colour of the precipitate produced by $AgNO_3$ in the solution made acid with HNO_3, and its behaviour with AmHO, though less trustworthy than the above tests, serve to indicate which of these acid-radicles is present; $AgCl$ being *pure white* and very easily soluble in AmHO, $AgBr$ being *pale yellow* and not readily soluble in AmHO, whilst AgI is *primrose yellow* and almost insoluble in AmHO. The action of hot strong H_2SO_4 on AgI, which is described in paragraph (265), is also distinctive.

DETECTION OF A CHLORIDE, BROMIDE AND IODIDE.

272.* The method of examination varies according to whether an iodide is present or absent. Since a chloride cannot be detected in the presence of an iodide, the latter must be separated before proceeding to examine for the chloride (273. 1). In paragraph (274) a process is given for detecting an iodide and bromide; it is of value only when a chloride has not to be tested for.

273.* To a small portion of the solution, made just acid if necessary by the addition of dilute H_2SO_4, add a little cold freshly prepared starch solution; then add drop by drop strong HNO_3, or one of the reagents mentioned in (269); the appearance of a dark blue coloration will show *the presence of an iodide.* The remainder of the solution is examined for a chloride and bromide by the directions given under 1 or 2, according as an iodide is present or absent.

1. *An iodide is present.*—Add to the solution a reagent consisting of a mixture of $CuSO_4$ solution with half its measure of strong H_2SO_3 solution and warm gently for a short time, Cu_2I_2 will be precipitated (266). In order to be sure that the iodide is entirely precipitated, a small quantity of the liquid must be filtered and warmed with a little more of the above reagent: if any further precipitate is caused, the filtered portion is returned to the unfiltered liquid, more of the reagent is added to the whole, which is then warmed for a time and again tested as above to see if the precipitation is complete; these trials are repeated until a small portion of the filtrate gives no further precipitate when it is warmed with more of the reagent.

The whole is then filtered until it is perfectly clear, KHO is added in excess to the filtrate and the liquid is boiled. The precipitate thus produced is filtered off and the filtrate, now freed from iodide, is tested for bromide and chloride as is directed below (2).

2. *Iodide is absent.*—Make the solution alkaline, if it is not already so, by the addition of pure KHO and evaporate it to dryness in a porcelain dish; scrape the residue off the sides of the dish and mix it with three or four times as much powdered $K_2Cr_2O_7$ by rubbing the two substances together in the dish with a pestle (260). Transfer this mixture into a tube or flask (page 138), taking the greatest care to let none of the powder enter the delivery-tube. Then pour in sufficient strong H_2SO_4 to cover the powder to the depth of about a quarter of an inch, at once insert the cork and dip the end of the delivery-tube into water contained in a test-tube. Mix the acid and powder by cautious shaking and warm the mixture gently, carefully guarding against allowing any of the mixture to get into the delivery-tube and thence into the water in the test-tube.

As soon as no more reddish brown vapour is visible in the delivery-tube, discontinue the heating and immediately withdraw the test-tube. If no coloured vapour has been evolved and the water in the test-tube is colourless, chloride and bromide are absent. If the water is coloured, pour into it sufficient CS_2 to form a layer at the bottom of the tube about as large as a small nut; close the mouth of the tube with the thumb and shake vigorously: if the CS_2 on settling shows a brown colour, the *presence of Br, derived from a bromide*, is shown.

Separate the water from the CS_2 by pouring it through a wet filter, add to the filtrate AmHO in excess, then $H\bar{A}$ in excess and $Pb\bar{A}_2$ solution. A yellow precipitate indicates the presence of a chromate, which proves indirectly that a *chloride was present* (260).

273*a*.* The above method will also serve for the examination for chloride, bromide and iodide in the precipitate obtained by adding $AgNO_3$ in excess to a solution. The precipitate is dried, and then fused for about ten minutes in a porcelain crucible with three or four times its bulk of fusion mixture; the cool mass is boiled with H_2O for some time and filtered, and the filtrate is examined as is directed in (273).

274.* *If only an iodide and a bromide* have to be tested for, pour sufficient CS_2 into the solution to form a large drop at the bottom; add drop by drop dilute chlorine water, or acidified $CaCl_2O$ solution, shaking thoroughly after each addition: iodine will be liberated if an iodide is present and will colour the CS_2 *purple*, but on continuing the addition of Cl this colour will be destroyed; the presence of a bromide may then be detected on the further addition of Cl by a *brown* coloration of the CS_2, which also disappears if too much Cl is added.

GROUP V.—PHOSPHATE GROUP.

Phosphates and arsenates resemble one another closely in many of their reactions; they present points of difference, however, which render their distinction possible.

PHOSPHATES ($-'''PO_4$).—Use $Na_2HPO_4.12H_2O$.

275. *$MgSO_4$* solution, to which some AmCl has been added and then a little AmHO, gives with the solution of a phosphate a white crystalline precipitate ($MgAmPO_4.6H_2O$): this precipitate is almost insoluble in AmHO, but is easily soluble in acids. If very little phosphate is present, the precipitate often appears only when the liquid has been gently warmed, then well stirred or shaken and allowed to stand for some time.

276. *Fe_2Cl_6* solution, if dropped in after the addition of a little $H\bar{A}$ and $Na\bar{A}$ solution, gives a yellowish white precipitate of $Fe'''PO_4$: the precipitation is hastened by *gentle* heat.

277. A drop of phosphate solution when added to a solution of $AmHMoO_4$ in HNO_3 gives a yellow precipitate: the formation of the precipitate is hastened by *gently* warming and stirring or shaking the liquid, but it often appears only after a time: a part of it usually adheres firmly to the inside of the tube, and may be removed by KHO, NaHO, or AmHO solution, in which it is readily soluble; it is only slightly soluble in inorganic acids, and is practically insoluble in HNO_3.

This test if properly performed is most delicate, but careful attention must be paid to the following precautions.

The $AmHMoO_4$ solution must be prepared according to the directions given in (552, remark 40).

The solution to be tested must not be alkaline to test paper, it is best made distinctly acid with HNO_3; it is then to be added *in small quantity only* to some of the $AmHMoO_4$ solution in a test-tube, more being added if no yellow precipitate forms after a few minutes when the liquid has been *gently* warmed and stirred.

This last precaution is extremely important, since *an excess of phosphate altogether prevents the formation of the precipitate.*

Show that this is the case by pouring a few drops of $AmHMoO_4$ solution into some Na_2HPO_4 solution acidified with HNO_3; no precipitate will form even on heating and shaking the liquid, since the phosphate is present in large quantity as compared with the $AmHMoO_4$; but on adding a few drops of this liquid to some fresh $AmHMoO_4$ solution the precipitate will appear.

It must also be remembered that HCl retards or prevents the formation of this precipitate; a solution in nitric acid should therefore always be used when possible.

278. *$AgNO_3$:* yellow precipitate (Ag_3PO_4): pour off a portion and show that the precipitate is soluble in AmHO and in HNO_3.

278*a*. A very delicate test for the presence of P in the form of a phosphate or otherwise, consists in strongly heating the dry solid substance with a small piece of Na in a little tube of hard glass closed at one end: on breaking the tube and breathing upon the powdered substance a smell of onions (H_3P) will be perceived.

If a phosphate is made into a paste with strong H_2SO_4 and heated strongly at the tip of the inner blowpipe flame, it gives a bluish green colour to the flame.

Many phosphates when they are moistened with $Co(NO_3)_2$ and heated in the outer blowpipe flame become blue.

Hydrogen phosphate, or phosphoric acid (H_3PO_4), is a colourless crystalline substance; its solution is strongly acid, but differs from H_2SO_4 by not charring paper which has been dipped in it and dried by heat; its presence may be confirmed by reactions (275, 277).

ARSENATES ($-'''AsO_4$).—Use $Na_2HAsO_4.12H_2O$ solution.

279. The precipitates formed in reactions (275, 276, 277) with a phosphate are precisely similar in appearance and general properties to those formed when the same reagents are added to an arsenate. The precipitate, however, which $AgNO_3$ gives with an arsenate is *brown*, whilst that given by a phosphate is *yellow;* also $AmHMoO_4$ gives a precipitate with an arsenate *only on boiling* the liquid and not by *gently heating* it as with a phosphate.

280. *The following differences serve to detect and separate a phosphate and an arsenate:—*

1. The fact that an arsenate yields a precipitate with $AmHMoO_4$ only when the liquid is *boiled*, whereas the corresponding precipitate with a phosphate is produced by *a gentle heat.*

2. An arsenate solution which has been boiled with strong HCl gives, when H_2S is passed into the hot liquid, first a white precipitate of S and then yellow As_2S_3; a phosphate solution under the same circumstances gives no precipitate.

3. In a *perfectly neutral* solution $AgNO_3$ gives with an arsenate solution a *brown* precipitate, and with a phosphate a *canary yellow* precipitate.

281. *The simplest method of ascertaining which of these two acid-radicles is present* is to throw the precipitate, produced on addition of AmCl, AmHO and $MgSO_4$, upon a filter, wash it with a little cold water and drop $AgNO_3$ upon it: if the precipitate consisted of $MgAmAsO_4$ it will become *brown*, if it was $MgAmPO_4$ it will become *canary yellow.*

Or the precipitate produced by the addition of AmCl, AmHO and $MgSO_4$ may be examined for arsenate by dissolving a part of it in dilute HNO_3, adding $AgNO_3$ as long as it causes any white precipitate and then very dilute AmHO gradually drop by drop; if arsenate is present a *brown* precipitate will form just before the liquid becomes alkaline to test paper: a phosphate will give a *yellow* precipitate. Instead of mixing the dilute AmHO with the acid liquid it may be gently poured in upon the top of it, when the colour will appear at the surface of contact.

DETECTION OF AN ARSENATE AND A PHOSPHATE.

282.* AmCl, AmHO and $MgSO_4$ are added as long as any further precipitate is caused: a small portion of the precipitate is then filtered off and tested at once for an arsenate by dropping $AgNO_3$ solution upon it. If the precipitate turns yellow, a phosphate alone is present. If the precipitate turns brown an arsenate is present and must be separated from the other part of the precipitate before testing for a phosphate.

The rest of the precipitate is accordingly filtered off and dissolved in a little boiling strong HCl; H_2S is then passed for several minutes into this hot solution, when the presence of an arsenate is confirmed by the precipitation of white S and yellow As_2S_3: the precipitate is filtered off and H_2S is again passed into the boiling filtrate; if any further precipitate is produced, the gas must be further passed until it no longer causes a precipitate; the clear filtrate is then evaporated just to dryness dissolved in a little dilute HNO_3 and tested for a phosphate by adding a few drops of it to $AmHMoO_4$ solution and gently warming the liquid (277).

An arsenite may be tested for by acidifying the filtrate from the Mg precipitate and passing H_2S into it: yellow As_2S_3 will be precipitated at once if an arsenite is present.

VI.—UNGROUPED ACID-RADICLES.

The following acid-radicles present no marked resemblances to one another or to other acid-radicles in their reactions; they are therefore usually detected by special tests.

BORATES ($-'BO_2$, or $-'''BO_3$).—Use $Na_2B_4O_7.10H_2O$.

283. Powder some borax finely and stir it with a little dilute HCl on a watch glass; immerse the lower half of a strip of turmeric paper in this liquid and dry it on a watch glass in the steam-oven or on a water-bath, or above a small flame: the part which was moistened will appear *reddish brown*, and will become *bluish black* when moistened with AmHO.

284. Pour methylated spirit upon some finely powdered borax in a test-tube or porcelain dish, add a little strong H_2SO_4, then heat the mixture and kindle the spirit; the flame will show a green edge, which is often best seen by blowing the flame out after it has burnt for a time and then relighting it. This flame coloration is similar to that given by certain other substances, but its spectrum, consisting of three or four equidistant green lines, is quite characteristic.

285. If a mixture of CaF_2 and $KHSO_4$ with borax or boric acid is finely powdered, moistened, and heated on a loop of platinum wire in the inner blowpipe flame, it colours the outer flame momentarily *green* after being heated for a short time. This test is not always decisive, since many phosphates and copper salts give a similar result; it is also liable to be masked by the flame colorations of other substances.

Hydrogen borate, or boric acid (HBO_2), is a white porous mass; when combined with $2H_2O$ it forms a crystalline scaly hydrate; these substances when strongly heated yield a fusible glass consisting of B_2O_3. The solution of the acid turns litmus paper wine red. Reactions (283, 284, 285) serve to detect boric acid. The acid is soluble both in H_2O and in alcohol; on evaporating these solutions, the acid partially escapes with the vapour of the liquid.

CHROMATES ($-''CrO_4$).—Use $K_2Cr_2O_7$.

All chromates are more or less yellow or red in colour. The yellow solution of a normal chromate becomes orange red on the addition of an acid, owing to the formation of a dichromate: while the reddish solution of a dichromate becomes pale yellow when made alkaline, owing to its conversion into a normal chromate.

286. Various substances which readily combine with oxygen, deoxidise acidified $K_2Cr_2O_7$ solution with production of a chromic salt, the colour of the solution at the same time changing from *orange red* to *bright green.*

Show this by adding HCl or H_2SO_4 to some $K_2Cr_2O_7$ solution, and deoxidising each of five portions by one of the following methods: in every case the colour of the liquid will change to bluish green:—

a. Pass H_2S into the hot solution; white S separates, owing to oxidation of the hydrogen.

b. Add $NaHSO_3$ or H_2SO_3 and warm.

c. Add alcohol and boil: the smell of aldehyde will be noticed.

d. Boil for some time, after adding much strong HCl: Cl gas will be slowly evolved.

e. Add Zn and warm: the action of the nascent H will be slow in this case.

The following equations show the reactions which occur with H_2S and with HCl:—

$$K_2Cr_2O_7 + 3H_2S + 8HCl = Cr_2Cl_6 + 2KCl + 7H_2O + S_3.$$
$$K_2Cr_2O_7 + 14HCl = Cr_2Cl_6 + 2KCl + 7H_2O + 3Cl_2.$$

The equations representing the other processes of deoxidation may be drawn out by the student.

The green solution resulting from the above reactions will give all the reactions for a chromic salt (102–104): and since chromic acid is reduced by boiling HCl and by H_2S to chromic salt, the presence of a chromate will lead to the precipitation and detection of $Cr_2H_6O_6$ in the ordinary course of analysis.

286 *a*. *Hydrogen peroxide* (H_2O_2), *or barium peroxide* (BaO_2), if added to a *cold* acidified solution of a chromate, produces an intense blue coloration, which is very evanescent: it is far more permanent if ether is first added and the liquid is vigorously shaken immediately after the addition of the peroxide, when the ether assumes and retains a blue colour; addition of a few drops of strong HNO_3 is useful. This reaction is extremely delicate and characteristic.

287. *$AgNO_3$: purple red* precipitate (Ag_2CrO_4): soluble in AmHO and in HNO_3.

288. *$Pb\bar{A}_2$: yellow* precipitate ($PbCrO_4$): soluble in KHO, insoluble in $H\bar{A}$.

289. *$BaCl_2$:* yellowish white precipitate ($BaCrO_4$): insoluble in $H\bar{A}$, soluble in HCl.

Hydrogen chromate, or chromic acid (H_2CrO_4?), is present in the reddish yellow liquid produced by dissolving CrO_3 in water and by acidifying a solution of a chromate. Its solution evolves Cl and becomes green if boiled with HCl; when neutralised by an alkali it becomes yellow and yields the above reactions for a chromate.

SILICATES ($-''SiO_3$).—Use Na_2SiO_3 solution, and finely ground sand (SiO_2) for a solid.

290. *HCl* added drop by drop to a solution of a silicate whilst stirring or shaking it, will give a gelatinous precipitate of H_4SiO_4, unless the solution is very dilute; the silicic acid, however, frequently remains in solution, hence the non-appearance of a precipitate is no proof of the absence of a silicate.

The solution of silicate used for these reactions is made of such a strength that acids do not cause a precipitate in it; but by boiling some of it down to a quarter of its volume before adding the acid the precipitate is usually obtained.

291. If a silicate is present in small quantity, or in dilute solution, it may be detected by acidifying the solution with HCl, then evaporating it to dryness in a porcelain dish, and heating the residue to 100° as long as acid fumes come off: insoluble SiO_2 is thus produced, and will remain undissolved as a white powder when the dry residue is stirred and warmed with strong HCl.

During the last part of the evaporation over a flame, after the silica has separated as a gelatinous mass, spirting will occur unless the heat is lessened and the residue is stirred with a glass rod: this is best avoided by finishing the evaporation upon a water-bath as soon as the liquid thickens. The SiO_2 which is left undissolved by HCl, is seen on stirring the liquid and at once pouring it out into a glass vessel; it is soluble in hot KHO solution.

292. *AmCl, or* Am_2CO_3, causes a gelatinous precipitate of H_4SiO_4, which usually forms only after a time.

293. If solid SiO_2 or a silicate is fused into a bead of Na_2CO_3, it causes frothing which is due to the evolution of CO_2:—$Na_2CO_3 + SiO_2 = CO_2 + Na_2SiO_3$.

294. When SiO_2 is fused into a bead of $NaAmHPO_4$ it is not dissolved, but floats about in transparent particles; and it is visible as little opaque masses in the cold bead. The particles of SiO_2 are usually most easily visible whilst they are in motion in the fused bead.

Certain silicates, if moistened with $Co(NO_3)_2$ and heated on charcoal in the outer blowpipe flame, become blue.

Hydrogen silicate, or silicic acid (H_4SiO_4), can exist in solution in water, and has a slightly acid reaction: it is ordinarily met with, however, as a colourless gelatinous substance which does not affect the colour of litmus, and is insoluble in water, very slightly soluble in acids, but dissolves as an alkaline silicate in solutions of the alkalis or alkaline carbonates. When strongly heated it yields a white insoluble powder (SiO_2), which gives reactions (293) and (294).

FLUORIDES (– 'F).—Use finely powdered CaF_2.

The ordinary tests for a fluoride depend upon the liberation of hydrofluoric acid, which is allowed to act upon a glass surface and to *etch* or corrode it.

295. *If strong H_2SO_4 is* warmed with a little *finely powdered* CaF_2 in a test-tube it liberates HF; this acid etches the glass and causes the sides of the tube to throw off the liquid when it is shaken up, as a greasy surface throws off water.

On washing away the cooled mixture and perfectly drying the inside of the tube, it will be found to be dimmed and roughened: gentle friction with a glass rod will often detect the rough surface even when it is not perceptible to the eye.

The etching, if slight, is best seen when the surface is breathed upon, or when the glass is so held as to observe its surface by the light it reflects.

296. The test is much more delicate if it is carried out as follows.

A watch glass is carefully heated by holding it in crucible tongs at some distance above a flame; its convex surface is then rubbed with a piece of paraffin wax, and is thus covered with a melted layer; this layer may be made uniform by once more heating the glass, allowing any excess of wax to drop off, and keeping the glass moving whilst it cools; the coating is finally hardened by immersion for a short time in cold water.

Lines or characters are then traced on the glass towards its centre by gentle pressure with the point of a penknife, the object being simply to remove the wax from these parts and not to scratch the glass.

The glass thus prepared is placed with its coated surface downwards as a cover to a platinum crucible or small leaden cup, which contains a little finely powdered CaF_2 and some strong H_2SO_4. After pouring some cold water into the glass to keep it cool, the bottom of the vessel is heated by a very small flame, and the covered vessel is allowed to stand for about fifteen minutes.

The watch glass is then removed, and cleansed by holding it with crucible tongs above the flame and quickly wiping off the melted wax: on the clean bright surface thus obtained the traced lines will usually be visible at once on holding the glass up to the light, but if mere traces of a fluoride were present the tracing will become visible when the cold surface is breathed upon.

297. The above methods do not serve for the detection of a fluoride in the presence of a silicate or of silica, since under these circumstances the action of H_2SO_4 liberates SiF_4 instead of HF.

SiF_4 possesses no power of etching glass, but it produces white fumes in moist air, and when it is passed into dilute AmHO it yields a colourless flocculent precipitate of H_4SiO_4 and produces AmF in the solution: the formation of this precipitate is sufficient proof of the presence of a fluoride; but after it has been filtered off the AmF may also be detected in the filtrate by adding $CaCl_2$, filtering off the precipitate of CaF_2, and drying and examining it by (296).

The above method is sometimes employed for the detection of a fluoride after intimately mixing SiO_2 with the substance to be tested.

Use for this purpose a mixture of CaF_2 and SiO_2 and drop a small piece of marble into the tube in which the SiF_4 is being evolved, in order to produce CO_2 which will carry that gas over into the AmHO solution. The changes which occur are thus expressed by equations:—

$$2CaF_2 + SiO_2 + 2H_2SO_4 = SiF_4 + 2H_2O + 2CaSO_4.$$
$$SiF_4 + 4AmHO = H_4SiO_4 + 4AmF.$$

298. *$CaCl_2$* when added to the solution of a fluoride gives an almost transparent gelatinous precipitate (CaF_2), which becomes more visible when the liquid is heated or when AmHO is added: it is slightly soluble in HCl, but almost insoluble in $H\bar{A}$.

Solution of NaF may be prepared for this reaction by fusing a mixture of powdered CaF_2 and fusion-mixture on *platinum* foil for several minutes; then boiling the cool mass on the foil in water, filtering, and acidifying the filtrate with $H\bar{A}$.

Hydrogen fluoride, or hydrofluoric acid (HF), is at ordinary temperatures a colourless gas which fumes in the air, etches a moist glass surface and dissolves easily in water. The solution of HF is acid in reaction and differs from all other acids by dissolving SiO_2.

FLUOSILICATES ($-''SiF_6$).—Use H_2SiF_6.

299. *$BaCl_2$:* white precipitate ($BaSiF_6$) which usually appears only on gently warming the solution, and differs in this respect from $BaSO_4$: it differs from $BaSeO_4$ (493. 7) by being only slightly soluble in boiling HCl; it is insoluble in alcohol. Allow this precipitate to settle and reserve it for reaction (301).

The above precipitate somewhat resembles $BaSO_4$ by being rather insoluble in HCl, but it differs in appearance by being more or less transparent and crystalline: but fluosilicates differ also from sulphates by giving no precipitate with $Sr(NO_3)_2$ or with $Pb(NO_3)_2$; further when heated with excess of AmHO they yield a flocculent precipitate of H_4SiO_4 and AmF in solution (297); fluosilicates differ also from sulphates by the following very distinctive reactions.

300. *KCl* solution when warmed with the solution of a fluosilicate gives a very transparent gelatinous precipitate (K_2SiF_6), which is only visible after it has been allowed to settle for a time; it is insoluble in alcohol.

301. If strong H_2SO_4 is added to H_2SiF_6 solution, or to a fluosilicate, and the mixture is heated, HF is given off and may be detected by its etching action on glass (296).

This reaction is especially useful for ascertaining whether a precipitate produced by $BaCl_2$ consists of or contains $BaSiF_6$. For this purpose the precipitate from (299) is allowed to settle; almost all the water is then poured off, the remainder is shaken up with the precipitate and at once poured into a watch glass; the rest of the water is then decanted, and the precipitate which remains on the glass is warmed for some time with strong H_2SO_4; on washing and drying the glass, it will be found to be corroded (295).

When the more delicate process (296) is employed, the precipitate should be shaken up with the liquid and poured off into a platinum crucible or leaden cup; the liquid is then decanted, and the precipitate is warmed with strong H_2SO_4 as is described in (296).

The processes of filtering off and drying the precipitate may be substituted for decantation, but they are less rapidly executed.

Hydrogen fluosilicate, or hydrofluosilicic acid (H_2SiF_6), is a strongly acid liquid, which volatilises entirely as $2HF + SiF_4$ when it is heated in a platinum vessel: if this acid is evaporated in a watch glass, the glass is etched: it is precipitated by KCl and by $BaCl_2$ (299, 300).

REACTIONS OF ORGANIC ACID-RADICLES.

The following acid-radicles, although usually classed amongst organic compounds, are included here because they are of frequent occurrence and their detection is usually a matter of little difficulty.

VII.—CYANIDE GROUP.

The simple and complex cyanides contain a group of elements (CN) known as cyanogen. They yield precipitates with $AgNO_3$, and evolve HCN when they are heated with dilute H_2SO_4. Many of their salts also evolve NH_3 when they are heated in the solid state.

CYANIDES (– 'Cy).—Use KCy solution.

The alkaline cyanides smell faintly of bitter almonds, owing to the escape of HCy.

303. *$AgNO_3$:* white precipitate (AgCy), most easily obtained by pouring a drop of the KCy solution into the $AgNO_3$ solution: show with three separate portions that this precipitate is soluble in KCy solution added in excess and in AmHO, but insoluble in dilute HNO_3.

If some of this precipitate is filtered off and well washed with hot water until HCl causes no milkiness with the last few drops of the washing water, then dried, scraped from the filter into a porcelain crucible and heated strongly, Ag is left: if this residue when cool is warmed with a little dilute HNO_3, it dissolves giving off red fumes, and a drop of HCl added to this solution gives a curdy precipitate of AgCl.

This reaction distinguishes AgCy from AgCl, AgBr and AgI, which like it are precipitated by $AgNO_3$ and are insoluble in HNO_3, since these when dried and strongly heated, fuse without undergoing decomposition; hence HNO_3 cannot dissolve out Ag, and HCl added to the HNO_3 gives either no precipitate or a mere milkiness, due to the imperfect washing of the precipitate.

This method is somewhat tedious and need seldom be used, cyanides being readily detected by the following reactions.

304. If some $FeSO_4$ solution is mixed with several drops of Fe_2Cl_6 and added to a solution of a cyanide, and the liquid is then made strongly alkaline with KHO and boiled, then cooled and acidified with HCl, *Prussian blue* ($Fe'''_4\overline{Cfy_3}$) is formed; this will appear as a deep blue precipitate, or if the quantity of cyanide present was small, it may remain dissolved giving a bluish green solution.

If no cyanide had been present, the addition of HCl would have produced an almost colourless and perfectly clear liquid.

305. If dilute H_2SO_4 is added to a cyanide contained in a test-tube fitted as is shown on page 138, and then a small piece of marble is dropped in and the liquid is boiled, HCy will be carried over by the CO_2 gas: if the gas is allowed to bubble through some Am_2S solution, AmCyS will therefore be formed.

This substance gives with Fe_2Cl_6 a *blood red* coloration (315), which may be obtained by boiling the Am_2S solution in a porcelain dish and acidifying it when cool with HCl; on adding several drops of Fe_2Cl_6 the red colour is well seen against the interior of the white dish; this proves that HCy has passed into the Am_2S, and therefore detects the presence of a cyanide.

A little of this red liquid should be added to some $HgCl_2$ solution; if the red colour is destroyed it was certainly due to the presence of a sulphocyanide.

On adding HCl to the Am_2S in the above reaction, white S will separate, unless the yellow liquid has been previously boiled in a porcelain dish until it becomes colourless, filling in distilled water as it evaporates; but this separation of S is advantageous, since its whiteness helps to show up even a faint reddish tint in the liquid.

The marble is added to cause evolution of CO_2 gas, and thus lessen the risk of the Am_2S being sucked back during the reaction.

306. The preceding test can often be more simply performed by boiling the solution of the cyanide in a porcelain dish with Am_2S, which must be added until the yellow colour remains after the liquid has been boiled for a short *time*: if a dark-coloured precipitate forms, this must be allowed to settle or be filtered off, before the colour of the filtrate can be seen. The liquid is then filtered if necessary and acidified with HCl in the porcelain dish; on adding Fe_2Cl_6 in small quantity a blood red colour will be produced, which is not removed by heat or by the addition of a little dilute HCl; it is at once destroyed, however, when a few drops of the liquid are added to some $HgCl_2$ solution.

Methods (304, 305, 306) are exceedingly delicate tests for a cyanide: (305) and (306), if less readily performed than (304), exceed it in delicacy. Method (306) is the more easy of the two to perform, and (305) is only used in cases where, after boiling with excess of Am_2S, the filtrate is dark-coloured and therefore the red coloration would be invisible.

$HgCy_2$ does not show the reactions for a cyanide by methods (303, 304, 305); but by boiling with Am_2S according to method (306), AmCyS is obtained in the filtrate from the black HgS precipitate, and will give the red coloration with Fe_2Cl_6. Solid $HgCy_2$ when heated in a small tube closed at one end also gives off cyanogen gas, which burns with a peach-blossom coloured flame.

Hydrogen cyanide, or hydrocyanic or Prussic acid (HCy), is a colourless, volatile, inflammable liquid, which smells like bitter almonds: it is *highly poisonous* when it is inhaled as vapour or swallowed as liquid: it readily mixes with water. The acid is easily detected by the tests already given for a cyanide.

FERROCYANIDES ($-''''FeCy_6$).—Use $K_4FeCy_6.3H_2O$.

307. *Fe_2Cl_6:* dark blue precipitate of *Prussian blue* ($Fe_4\overline{Cfy}_3$) *: insoluble in HCl, soluble in $H_2C_2O_4$ to a dark blue liquid, and changed by KHO into brown $Fe_2H_6O_6$.

The solubility in $H_2\overline{O}$ and insolubility in HCl are shown by pouring off portions, heating them with HCl and $H_2\overline{O}$ respectively, filtering and noting that the filtrate is blue.

308. *$FeSO_4$:* light blue precipitate ($Fe''K_2\overline{Cfy}$), which becomes darker in colour by oxidation on exposure to the air, or on addition of Cl- or Br-water, or when warmed with HNO_3: it is insoluble in HCl.

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*See abbreviations on page 75.

309. $CuSO_4$*:* a chocolate coloured precipitate ($\text{Cu}''_2\overline{\text{Cfy}}$), insoluble in $\text{H}\bar{\text{A}}$.

310. $AgNO_3$*:* white precipitate ($\text{Ag}_4\overline{\text{Cfy}}$), insoluble in HNO_3 *and in AmHO:* when heated with HNO_3 this precipitate changes to orange red $\text{Ag}_6\overline{\text{Cfy}_2}$, which is *soluble in AmHO*.

FERRICYANIDES $[-{}^{\text{vi}}(FeCy_6)_2]$.—Use $\text{K}_6(\text{FeCy}_6)_2$.

311. Fe_2Cl_6*:* dark green or brown coloration; no precipitate is formed, as may be seen by adding much water to the liquid.

312. $FeSO_4$*:* dark blue precipitate of *Turnbull's blue* ($\text{Fe}''_3\overline{\text{Cfy}_2}$): insoluble in acids: blue colour destroyed by KHO.

313. $CuSO_4$*:* yellowish precipitate.

314. $AgNO_3$*:* orange coloured precipitate ($\text{Ag}_6\overline{\text{Cfy}_2}$), insoluble in HNO_3, *soluble in AmHO.* Frequently a white residue of $\text{Ag}_4\overline{\text{Cfy}}$ is left on adding AmHO, showing that $\text{K}_4\overline{\text{Cfy}}$ was present in the $\text{K}_6\overline{\text{Cfy}_2}$ solution.

SULPHOCYANIDES (– 'CyS).—Use KCyS solution.

315. Fe_2Cl_6*:* blood red coloration, but no precipitate: the colour is weakened but not destroyed by HCl, but disappears when the liquid is dropped into HgCl_2 solution.

316. Ferro- and ferri-cyanides can be found and distinguished by their first three tests, or by the precipitate given by AgNO_3 in the solution acidified with HNO_3; this precipitate will have a more or less decided orange red colour if a ferricyanide is present; on filtering or decanting, and warming the precipitate with AmHO, white $\text{Ag}_4\overline{\text{Cfy}}$ will be left if a ferrocyanide was present, and on acidifying the filtrate with HNO_3 orange red $\text{Ag}_6\overline{\text{Cfy}_2}$ will be reprecipitated.

VIII.—OXALATE GROUP.

The acids and salts containing the three following acid-radicles differ from the foregoing by being charred and emitting a smell of burning when heated in the solid state; with oxalates, however, these effects are scarcely perceptible.

If the salt contains K, Na, Ba, Sr, or Ba, the residue left after ignition will contain a carbonate which will effervesce with an acid, whereas the original unignited substance would not do so.

OXALATES ($-''C_2O_4$).—Use $Am_2C_2O_4.2H_2O$.

317. *Strong H_2SO_4* heated with a solid oxalate causes an effervescence due to the evolution of a mixture of CO and CO_2:—

$$Am_2C_2O_4 + 3H_2SO_4 = CO + CO_2 + 2AmHSO_4 + H_2SO_4.H_2O.$$

The CO is detected by burning with a blue flame when a light is applied, and the CO_2 by rendering a drop of lime water on a glass rod milky.

No blackening or charring is produced by strong H_2SO_4; oxalates differ in this respect from other organic acids and their salts.

318. *$CaCl_2$ or $CaSO_4$ solution, or lime water*, added to the solution of an oxalate, gives a white precipitate ($Ca_2C_2O_4$), which is insoluble in AmHO and in $H\bar{A}$, but soluble in HCl and in HNO_3.

318 *a*. Filter off this precipitate and pour upon part of it on a watch glass dilute HCl, it will not effervesce; dry the remainder, and heat it to full redness on platinum foil, it is thus converted without blackening into $CaCO_3$, which effervesces with dilute HCl.

This is a general test for oxalate of K, Na, Ba, Sr, or Ca; since each of these, when it is ignited, leaves a *carbonate*, which effervesces with an acid.

319. An oxalate in the solid state, or in strong solution, when heated with MnO_2 and H_2SO_4 evolves CO_2 :—

$$Am_2C_2O_4 + MnO_2 + 3H_2SO_4 = 2CO_2 + MnSO_4 + 2AmHSO_4 + 2H_2O$$

Since commercial MnO_2 usually contains some carbonate, it will give off CO_2 when treated with H_2SO_4. If the MnO_2 is not known to be free from carbonate it is best to add a little H_2SO_4 to some of the MnO_2 in a test-tube, and heat to boiling; if CO_2 is found in the tube by a glass rod with a drop of lime water on its end, continue to boil, occasionally sucking fresh air into the test-tube through a glass tube pushed down near to the surface of the liquid; as soon as no CO_2 can be detected in the tube after boiling for a short time, the liquid being still distinctly acid, the $Am_2C_2O_4$ is added and the mixture again heated; CO_2 will be rapidly given off and may be detected by the ordinary methods.

Hydrogen oxalate, or oxalic acid ($H_2C_2O_4.2H_2O$), when heated in a glass tube closed at one end partly sublimes unaltered, and is partly decomposed evolving vapour which causes coughing; it yields reactions (317) and (318). The crystals dissolve readily in water to an acid poisonous liquid.

TARTRATES ($-''C_4H_4O_6$).—For a neutral tartrate use $KNa\bar{T}$, for an acid tartrate $H_2\bar{T}$ or $NaH\bar{T}$.

320. A solid tartrate when heated strongly in a tube chars, giving off a smell of burnt sugar.

321. A solid tartrate when heated with strong H_2SO_4 rapidly blackens, giving off a mixture of CO, CO_2 and SO_2.

322. *$AgNO_3$* produces in solution of a neutral tartrate a white curdy precipitate ($Ag_2\bar{T}$), soluble in AmHO and in HNO_3. Allow some of this precipitate to settle in a test-tube, and pour off as much of the clear liquid as possible; then pour *very dilute* AmHO drop by drop into the tube, until the precipitate, after being shaken up with the liquid, is nearly but *not quite* dissolved; then dip the test-tube into cold water in a beaker, which is slowly heated to about 60°; a brilliant mirror-like film of Ag will be *deposited* upon the interior of the tube.

If the liquid is quickly boiled, the Ag deposits as a dull black precipitate which does not adhere to the glass, and is not characteristic.

323. $CaCl_2$ precipitates from the solution of a neutral tartrate white or crystalline $Ca\bar{T}$, which often separates only after shaking the liquid well and letting it stand for a time; it is soluble in acids, and its formation is retarded by the presence of AmCl and other Am-salts. This precipitate when freshly formed is completely soluble in strong cold KHO solution, if this is free from K_2CO_3: on boiling the solution the $Ca\bar{T}$ is reprecipitated, but it dissolves again more or less completely when the liquid is cold.

Decant the clear liquid and pour upon the precipitate a little very dilute AmHO; then drop in a small crystal of $AgNO_3$ and warm carefully as in (322). A silver mirror will form on the part of the glass on which the $AgNO_3$ crystal rests.

324. *KCl, or better* $K\bar{A}$, if added to the solution of an acid tartrate, gives a white crystalline precipitate ($KH\bar{T}$), which forms slowly unless the liquid is vigorously stirred or shaken. If $H\bar{A}$ is added at the same time, the precipitate may also be obtained with neutral tartrates. The precipitate is soluble in alkalis and in mineral acids, but is almost insoluble in alcohol; hence alcohol is often added to hasten the precipitation.

325. If a few drops of Fe_2Cl_6 solution are added to the solution of a tartrate, which is then made strongly alkaline by addition of KHO or AmHO, no precipitate will appear.

The precipitation by alkalis of several other metals, such as Al, Mn, Cu, is similarly prevented by the presence of a tartrate: this is owing to the formation of a double tartrate, which is not decomposed by the alkali.

Hydrogen tartrate, or tartaric acid ($H_2\bar{T}$), occurs in colourless crystals which do not alter in the air: they taste pleasantly acid, and are soluble in water and in alcohol: they are detected by heating them alone or with strong H_2SO_4

(320, 321): if dissolved and neutralised they also yield the reactions in (322, 323).

ACETATES ($-'C_2H_3O_2$).—Use $Na\bar{A}.3H_2O$.

326. A solid acetate if strongly heated blackens and evolves the vapour of acetone, which possesses a peculiar smell and burns with a bright flame when kindled.

327. An acetate, when heated with strong H_2SO_4, evolves vapour of $H\bar{A}$, which in large quantity is very pungent, but in smaller quantity smells like vinegar. If the acetate is first mixed with alcohol, then with strong H_2SO_4, and the mixture is warmed, a fragrant smell of ethyl acetate ($Et\bar{A}$) is emitted; this vapour is most strongly smelt when the liquid is shaken after it has cooled for a short time.

328. Fe_2Cl_6 added in small quantity to the solution of an acetate causes a deep red coloration: if this red solution is made neutral but not alkaline with AmHO and then boiled, all the Fe is precipitated from it as light brown basic acetate and the liquid becomes colourless. The red colour of the solution before being boiled is changed to yellow by the addition of a few drops of dilute HCl, and is in this way distinguished from the coloration due to sulphocyanide (315).

Hydrogen acetate, or acetic acid ($H\bar{A}$), is known as *glacial acetic acid;* at temperatures below 17° C. it is a colourless crystalline substance which readily melts and emits when heated a very pungent and inflammable vapour: it is strongly acid, and is completely volatile without decomposition. Acetic acid dissolves in water readily, imparting its peculiar smell to the liquid in a degree depending upon its dilution.

The reactions for certain other organic acid-radicles are given at the end of the Sixth Section of this book.

SECTION V.

ANALYSIS OF SIMPLE SUBSTANCES, CONTAINING ONE METAL AND ONE ACID-RADICLE.

This section may be neglected by a student who is learning the full analytical course.

Two metals may occasionally be present, as when an alum or tartar-emetic [$K(SbO)\overline{T}$] is given for analysis; also there may be two acid-radicles in the liquids given for analysis, since a salt which is insoluble in water is occasionally dissolved in an acid: but the student should always be informed of this if it in any way complicates the analysis. It will not usually do so, since in the most commonly occurring case, that of a phosphate dissolved in HCl, the PO_4 is detected in Group III., and accordingly no other acid-radicle is looked for.

Examples of substances for analysis are given in paragraph (559).

The numbers in brackets refer to the paragraphs in the text, in which tests or processes to be employed by the student are fully described.

INTRODUCTORY REMARKS.

330. Before proceeding to detect the metal or acid-radicle in the solution of a simple substance by systematic analysis, it is best to make a few preliminary experiments (333–338*a*), which will generally give some idea as to what substance is present, and will occasionally detect with certainty the metal or acid-radicle, or both of them.

Even if the composition of the substance has been proved by this preliminary examination, it is usually best to proceed to the next process, which consists in endeavouring to dissolve the substance in water or acids (332), if it is not already in the liquid form: the addition to this solution of group-reagents in appropriate succession, will then determine the Analytical Group to which the metal present belongs (339).

Table showing the Analytical

<table>
<tr>
<td rowspan="4"><i>Note.</i>—It will be observed that there are two divisions of Groups II. and III. In analysis it is usual to separate the two divisions of Group II. from one another, after having separated the whole group from the remaining groups; the two divisions of Group III. are by some analysts precipitated together and subsequently separated; they are, however, often precipitated separately, and treated as distinct groups.</td>
<td>Metals whose chlorides are insoluble in water and dilute acids, and which are therefore precipitated from their solutions by hydrochloric acid.</td>
<td colspan="2">Metals whose sulphides are insoluble in dilute acids, and which are therefore precipitated from <i>acid</i> solutions by sulphuretted hydrogen.</td>
</tr>
<tr>
<td rowspan="2">GROUP I.
<i>Hydrochloric Acid, or Silver Group</i></td>
<td colspan="2">GROUP II.
<i>Sulphuretted Hydrogen Group</i></td>
</tr>
<tr>
<td>GROUP II.A.
<i>Copper Group</i>
Sulphides insoluble in NaHO and in Am_2S.</td>
<td>GROUP II.B
<i>Arsenic Group</i>
Sulphides soluble in NaHO and in Am_2S.</td>
</tr>
<tr>
<td>[Lead . . . Pb.]
Silver . . . Ag
Mercurosum . Hg'
1.</td>
<td>Mercuricum Hg''
[Lead . . Pb]
Bismuth . Bi
Copper . Cu
Cadmium . Cd
2.</td>
<td>Tin . . Sn
Arsenic . As
Antimony Sb
3.</td>
</tr>
</table>

In the preceding Table the metals are classified in their analytical groups, each group being headed by its distinctive number and name, and by the name of the group-reagent which serves to precipitate its members and to separate them from those of the succeeding groups.

After discovering to which of the above analytical groups the metal present belongs, it is identified by the properties or appearance of the group-precipitate; then its presence is confirmed, if necessary, by special tests made on the precipitate or on another portion of the *original solution** (344–348).

The acid-radicle, if it has not been already detected during the examination for the metal, is tested for in a less systematic manner. A few preliminary experiments are first performed (335–338*a*); and if these do not detect the acid-radicle, it is found partly by a systematic examination, which includes that described above for the detection of the metal, but more commonly by special tests (349–356).

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* By the *original solution* is meant the solution of the substance, or the substance itself, if a liquid is under analysis, to which no *reagent* has been added.

Classification of the Metals.

Metals which are precipitated as hydrates from their solutions containing ammonium chloride when made alkaline with ammonia, or as sulphides or hydrates when ammonium sulphide or sulphuretted hydrogen is subsequently added.				Metals whose carbonates are precipitated by addition of ammonium carbonate in the presence of ammonium chloride.		Metals whose solutions are not precipitated by any of the foregoing group-reagents in the presence of ammonium chloride.	
GROUP III. *Ammonia and Ammonium sulphide Group*				GROUP IV. *Ammonium carbonate or Barium Group*		GROUP V. *Potassium Group*	
GROUP III.A. *Ammonia or Iron Group*		GROUP III.B. *Ammonium sulphide or Zinc Group*					
Aluminium	Al	Zinc	Zn	Barium	Ba	Ammonium	NH_4
Iron	Fe	Manganese	Mn	Strontium	Sr	Potassium	K
Chromium	Cr	Nickel	Ni	Calcium	Ca	Sodium	Na
		Cobalt	Co			Magnesium	Mg
4.		5.		6		7.	

A substance which is insoluble in water and in acids is examined according to the special directions in (367–370).

Since in these analyses only one metal and one acid-radicle are to be looked for, the examination for the metal is carried no further when one metal has been detected with certainty, and the examination for the acid-radicle is similarly at an end when the presence of one such radicle has been satisfactorily proved. It must, however, be understood that substances given for analysis are liable to contain impurities, which will yield only slight indications; if in the course of analysis, therefore, *only a slight* result is obtained, proceed until some substance is detected in larger quantity, entering against the slight result, *Trace of* ——.

The separation and washing of a precipitate by *decantation* (30, 31), is recommended whenever it is possible.

In making the analysis each result must be fully entered in the note-book *as soon as the experiment has been performed*, the form of entry being the same as that adopted for the directions in the text.

DIRECTIONS FOR THE ANALYSIS OF A SIMPLE SUBSTANCE.

The substance given for analysis may be either a solid or a liquid. If it is a solid examine it by (331 *et seq.*) omitting (334), if a liquid is to be analysed begin at (334).

THE SUBSTANCE IS A SOLID.

331. Make a careful examination of the properties of the substance.

Note its colour. The following are some of the most commonly occurring coloured substances:—

Blue: hydrated cupric salts, anhydrous cobalt salts.
Yellow: normal chromates.
Brownish yellow: ferric salts, PbO.
Orange red: certain dichromates.
Red: HgO, HgI_2, HgS, Pb_3O_4.
Green: Cr_2O_3, $CuCl_2$, salts of Ni and Cr. *Pale green:* ferrous salt.
Pink: hydrated cobalt salts. *Pale pink,* manganous salts.
Dark brown: PbO_2.
Black: Sb_2S_3, CuO, MnO_2. FeS.
White: anhydrous salts of Cu and Fe″, and many other powdered substances.

Also try its smell, and observe whether it is in powder, crystals, or non-crystalline pieces: carefully write down any appearance or property which may strike you on examining the substance with the naked eye or with the assistance of a lens.

After noting down the results of this examination, the substance is to be powdered as finely as possible.

If the substance possesses metallic lustre or appearance, work as is directed under (366): if it is not metallic in appearance, proceed to ascertain its solubility by the directions contained in (332), and according to the result thus obtained, conduct the further analysis of the substance *by (333)* or by (367).

SOLUBILITY OF THE SUBSTANCE.

332. Since the method of examination of a solid substance depends upon whether it is soluble or insoluble in water or acids, the following experiments are tried.

Place a small quantity of the *finely powdered* substance in a broad test-tube or a boiling-tube, fill the tube about one-third with water, and heat to boiling:—

<table>
<tr>
<td rowspan="4">1. <i>The substance dissolves.</i>

After examining a portion of the solid substance by the preliminary tests (333) and (335–338 <i>a</i>) proceed to examine the colour and reaction of the liquid to litmus by (334, I., II.), and, refer to (330) for directions for analysis.</td>
<td colspan="4"><i>The substance does not dissolve:</i> heat a fresh portion with dilute HCl to boiling, if it does not dissolve decant and boil the residue with strong HCl:—</td>
</tr>
<tr>
<td rowspan="3">2. <i>The substance dissolves.</i>

<i>Absence of Group I., except possibly Pb.</i>

After examining a portion of the solid substance by the preliminary tests (333) and (335–338 <i>a</i>) note the colour of the HCl solution (334, I.), and proceed to examine it for the metal by (339).</td>
<td colspan="3"><i>The substance does not dissolve:</i> put aside the test-tube containing the substance with HCl, and stir a fresh portion of the substance with a little dilute HNO_3; if it is not dissolved, boil. If dilute HNO_3 does not dissolve it, heat with a little strong HNO_3:—</td>
</tr>
<tr>
<td rowspan="2">3. <i>The substance dissolves.</i>

After examining a portion of the solid substance by the preliminary tests (333) and (335–338 <i>a</i>) note the colour of the solution in HNO_3 (334, I.) and examine it for the metal by (339).

<i>Note.</i> — Before testing for Group II., add strong HCl and boil down considerably.*</td>
<td colspan="2"><i>The substance does not dissolve:</i> mix together the contents of the two tubes in which the substance was heated with HCl and with HNO_3, and heat the mixture:—</td>
</tr>
<tr>
<td>4. <i>The substance dissolves.</i>

After examining a portion of the solid substance by the preliminary tests (333) and (335–338 <i>a</i>) examine the solution in HNO_3 and HCl mixed for the metal by (339). See <i>Note</i>, column 3.</td>
<td>5. <i>The substance does not dissolve.</i>

Examine a fresh portion of the substance by (307 <i>et seq.</i>).</td>
</tr>
</table>

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* This method of proceeding removes HNO_3, which would cause with H_2S a deposit of S.

PRELIMINARY EXAMINATION OF A SOLUBLE SOLID.

333. The Table is thus arranged: in the first column is placed the experiment to be made; in the second a list of the results, any one or more of which may be observed; and in the third the inferences to be drawn from the results. Confirmatory or supplementary experiments are occasionally entered, they are, however, only to be tried when the result preceding them in the second column has been noticed.

A more complete preliminary examination will be found in (387–405).

Experiment.	Observation.	Inference.
I. Heat a portion of the substance in an ignition-tube, or in a small dry test-tube, first gently and at last to redness.	I. The substance does not change .	**Absence of volatile, fusible and organic substances, and of water.**
	II. The substance changes:—	
	1. Drops of water condense in the top of the tube.	**Presence of H_2O.**
	2. The substance fuses easily . . .	**Probable presence of a salt of Na or K, or of Ba, Sr, Ca, Mg**
	3. The substance sublimes	**Presence of a salt of NH_4, As, or Hg: possibly also of $H_2C_2O_4$, of S (yellow), and of I (purple vapour).**
Confirmatory. — Boil another portion of the substance with KHO solution.	A smell of NH_3 is evolved and white fumes appear when a rod moistened with strong HCl is held to the mouth of the tube.	**Presence of NH_4. Confirm by $NaH\bar{T}$ (53), and proceed to (335).**
Confirmatory. — Mix another portion of the substance with about three times as much powdered Na_2CO_3 and KCy dry and heat in an ignition tube.	A grey sublimate of minute metallic globules, best seen by a lens.	**Presence of Hg. Pour AmHO upon the original substance, if it blackens Hg is present as a mercur*ous* compound.**
	A black mirror-like sublimate . . .	**Presence of As.**
	4. The substance blackens, and gives off CO_2, which makes a drop of lime-water on a glass rod milky.	**Presence of an organic acid-radicle, probably $\bar{A}$, $\bar{O}$ or $\bar{T}$: the last is known by a smell of burnt sugar.**
	The cool residue in the tube effervesces with HCl, whereas the original substance did not.	**The organic acid-radicle is united with K, Na, Ba, Sr, Ca, or Mg.**

(*Continued on next page*).

Experiment.	**Observation.**	**Inference.**
	5. Gas or vapour is given off: smell the vapour:—	
	Red nitrous fumes, recognized by their smell.	**From nitrate of a heavy metal.**
	SO_2, known by its smell and by turning $K_2Cr_2O_7$ solution green.	**From combustion of free S or a sulphide; possibly also from a sulphate, sulphite, or theiosulphate.**
	Cl, Br, I, known by smell and colour: Br and I by action on starch paste, and Cl by bleaching moist litmus.	**From certain chlorides, bromides, or iodides, also free I.**
	A slip of wood with a spark at its end, glows brightly or bursts into flame.	**O from chlorate or nitrate, or N_2O from decomposition of NH_4NO_3.**
	The gas can be lighted and burns at the mouth of the tube; note the colour of the flame:—	
	***Pale greenish yellow;* the gas burns explosively, and nitrous fumes are seen.**	**Presence of NH_3, from strongly heated NH_4NO_3.**
	***Bright white,* the unburnt gas smells of garlic.**	**Presence of PH_3, probably from a hypophosphite.**
	***Peach-blossom coloured flame,* grey sublimate of Hg.**	**Presence of Cy, from $HgCy_2$.**
	6. The substance changes colour:—	
	Yellow, hot. *White,* cold.	ZnO
	Yellow brown, ,, *Yellow,* ,,	PbO, SnO_2 or Bi_2O_3
	Dark red or *brown,* ,, *Red brown,* ,,	Fe_2O_3
II. Dip into the powdered substance a moistened loop of platinum wire, and hold the loop in the Bunsen flame; moisten with strong HCl, and again hold in the flame.	I. The flame is not coloured . . .	Probable absence of the substances enumerated below.
	II. The flame is coloured:—	
	Intense yellow *	Presence of Na.
	*Pale lilac**, appearing *crimson red* through the indigo-prism . .	,, K.
	Red Appearing *green* through the indigo-prism . . .	,, Ca.
	Appearing *intense red* through the indigo-prism . .	,, Sr.
	Green *Yellowish green*	,, Ba.
	Bright green, with *blue* centre after moistening with HCl.	,, Cu.
	Bright green	,, B_2O_3.
	Blue *Pale blue, livid*	Presence of As, Sb, Pb.
	Vivid blue	,, $CuCl_2$.

*** If the coloration for Na or K is intense, no further examination for the metal is necessary but the presence of K should be confirmed by stirring with NaHT(48). Proceed to (335). If the coloration is slight, a trace only of the metal is present, and the examination for another metal is continued.**

Experiment.	Observation.		Inference.
III. Heat a portion of the substance in a cavity scooped on charcoal, in the blowpipe flame.	1. The charcoal "deflagrates," or burns rapidly.		Presence of a nitrate or chlorate.
	2. A white mass is left on the charcoal which does not fuse, and shines brightly when strongly heated.		Presence of Ba, Sr, Ca, Mg, Al, or Zn.
Confirmatory. — Detach a small portion of the white mass, place it on red litmus paper and moisten it with a drop of water.	The red paper becomes blue in a short time where the substance rests.		Presence of Ba, Sr, Ca, or possibly of Mg.
Confirmatory.—If the moist residue is not alkaline to test-paper, moisten the remainder on the charcoal with $Co(NO_3)_2$ solution and heat again strongly in the blowpipe flame.	A *blue* residue		Presence of Al, or possibly of a phosphate, silicate, or borate.
	A *pink* residue		Presence of Mg.
	A *green* residue		,, Zn.
	3. The residue is not white and does not consist of globules of metal, neither is there any incrustation formed upon the charcoal; proceed to Exp. IV.		
	4. The residue contains metallic globules or an incrustation forms on the charcoal; proceed to Exp. V., omitting Exp. IV.		
IV. Fuse a small quantity[*] of the substance into a clear colourless borax bead first in the inner then in the outer flame, noting in both cases the colour of the bead whilst hot and when cold.	I. The bead is colourless when heated in flames both.		Absence of the metals below.
	II. The bead is coloured :—		
	In outer flame.	In inner flame.	
	Green, hot; *blue*, cold.	*Red* or *colourless*	Presence of Cu.
	Blue, hot and cold.	*Blue*, hot and cold	,, Co.
	Brown or *dingy purple*, hot; *light brown*, cold.	*Grey* or *black*, hot and cold . .	,, Ni.
	Brown, hot; *yellow*, cold.	*Bottle green*, hot and cold . .	,, Fe[†].
	Green[†], hot and cold.	*Green*, hot and cold	,, Cr.
	Purple, hot and cold.	*Colourless*, hot and cold . .	,, Mn.
Confirmatory. — The presence of Cr or Mn may be confirmed by fusing a portion of the substance on platinum foil or wire with Na_2CO_3 and KNO_3.	A *yellow* mass on cooling		,, Cr.
	A *bluish green* mass on cooling . . .		,, Mn.

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[*] It is necessary to adjust carefully the quantity of substance fused into the bead; if too much is used, the bead will often appear opaque; if too little is employed, one of the metals may escape detection. It is best at first to fuse only a minute quantity into the bead, adding more if no colour, or but a faint colour, is produced.

[†] Cr if present as a chromate gives a bead *brown* whilst hot, after having been fused in the outer blowpipe flame; but this brown colour is not reproduced in the case of Cr when the bead, after becoming green by being heated in the inner flame, is again heated in the outer flame.

Experiment.	Observation.		Inference.
V. Mix the substance in a small cavity on wood-charcoal with Na_2CO_3 and KCy, and heat strongly in the inner blowpipe flame for several minutes. If metallic globules are obtained, detach one and strike it sharply on the bottom of an inverted mortar with the pestle, and note whether it is crushed to powder (*brittle*) or merely flattened out into a cake (*malleable*). If the globules are white and malleable, take one upon the point of a penknife and see if it will mark paper as a black-lead pencil does.	Metallic scales or globules are obtained, or an incrustation forms on the charcoal:—		
	Globules.	*Incrustation.*	
	White and *brittle.*	*White* . . .	Presence of Sb.
	White and *brittle.*	*Yellow* . . .	" Bi.
	Red and *malleable.*	*None* . . .	" Cu.
	White and *malleable* marking paper easily. . .	*Yellow* . . .	" Pb.
	White and *malleable* not marking paper, and readily fusible.	*None* . . .	" Sn.
	White and *malleable* not marking paper, fusible only with difficulty.	*None* . . .	" Ag.
	None. . .	*Yellow* whilst hot, *white* when cold; becomes green when moistened with $Co(NO_3)_2$ solution and reheated in the outer flame.	" Zn.
	None. . . .	*Brown* . . .	" Cd.
	None. . . .	*White;* on smelling the charcoal a smell of onions is perceived.	" As.

After the completion of the preliminary tests for the metal, a solution of the substance is made according to the directions in (332), and this solution is examined as is there directed.

334. Preliminary Examination of a Liquid.

Experiment.	Observation.	Inference.
I. Note the colour of the liquid. *Note.*—If the colour has to be observed by night it should be examined by the white light obtained by burning a piece of magnesium ribbon. Delicate tints are best seen by looking through some thickness of the liquid at a sheet of perfectly white paper.	I. The liquid is coloured:—	
	Pink Very delicate	Presence of Mn or dilute Co solution.
	Intense	Presence of Co.
	Green Very delicate	„ Fe'', or Ni or Cr in dilute solution.
	Intense	„ Ni, Cr, or Cu.
	Yellow Reddish yellow . . .	„ Fe''' or a bichromate.
	Light yellow . . .	„ neutral chromate.
	Blue	„ Cu.
	Violet or *purple* . . .	„ Cr or a permanganate.
	II. The liquid is colourless . . .	Absence of Co, Ni, Cr, Cu, &c.
II. Dip pieces of red and blue litmus paper into the liquid.	*It is neutral* . . . . . . (Not affecting the colour of either paper.)	Absence of free acids and alkalis, and of all salts but some of those of Am, Na, K, Mg, Ba, Sr, Ca, Ag.
	It is acid . . . . . . (Turning blue paper red. If, on adding Na_2CO_3 to a part of the solution and warming, no effervescence occurs, free acid and acid salt are absent.	Presence of a free acid, of an acid salt, or of a salt with acid reaction.
	It is alkaline . . . . . . (Turning red paper blue.) If the colour of the paper becomes only slightly changed it indicates the probable presence of an alkaline salt.	Presence of a hydrate of K, Na, Am, Ca, Sr, or Ba, or of a salt with alkaline reaction.

Experiment.	Observation.	Inference.
III. Evaporate (27) some of the liquid upon a thin watch glass* heated very gently by supporting it some inches above a small flame upon a piece of wire gauze, or by means of a sand or water bath. If a residue remains on the glass bring this gradually into the flame and heat it more strongly.	*A residue is left.* . . . . . On being strongly heated the residue blackens and emits a smell of burning . . The smell resembles that of burning sugar. *Note.*—If the cold residue after ignition effervesces with a drop of HCl, whereas the unignited residue did not, the organic acid-radicle is united with Ba, Sr, Ca, Mg, Na, or K. The results in Exp. I., par. 333, may be here observed and should be noted. Keep this residue for Exp. VI.	Presence of some dissolved solid. Pres. of $\overline{A}$, $\overline{O}$, or $\overline{T}$. Pres. of $\overline{T}$.
	No residue is left . . . . . If the liquid is also colourless, tasteless, and without action on litmus-paper, it must consist only of distilled water, and no further examination of it need be made. If the liquid is alkaline, NH_3 must be looked for; if it is acid, H_2CO_3, H_2SO_3, HCl, HNO_3, H_2SO_4, and other volatile acids only need be tested for.	Absence of any dissolved solid.
IV. Add excess of strong NaHO or KHO solution and boil (51).	A smell of NH_3; moist red litmus is turned blue, and a rod moistened with strong HCl produces white fumes when held in the mouth of the test-tube.	Presence of NH_4. No further test need be tried for a metal. Proceed to (335).
V. Experiment II. (page 183) is then tried by dipping the platinum wire into the liquid, concentrated, if necessary, by evaporation.		
VI. Experiment IV. (page 184) is tried by fusing some of the residue from Exp. III. (above) in the borax bead.		

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* A piece of thin glass from the side of a broken flask is a cheap and useful substitute for a watch glass.

Preliminary Examina-

335. Pour dilute H_2SO_4 in excess upon the substance in a test-tube,

336. A gas is evolved, shown by effervescence or by a characteristic

1. *Colourless gas without smell.*	2. *Gas smelling of burning sulphur.*	3. *Gas with a most fetid smell.*	4. *Yellow gas smelling of Cl.*
A drop of lime or baryta-water, held in the tube upon the end of a glass rod becomes milky :— *Presence of a carbonate*	A drop of $K_2Cr_2O_7$ solution held in the tube upon the end of a glass rod, becomes green :— *Presence of a sulphite.*	A slip of filter-paper moistened with alkaline solution of $Pb\bar{A}_2$, and held in the tube, is blackened :— *Presence of a sulphide.*	A slip of moist litmus-paper held in the tube is bleached :— *Presence of a hypochlorite.*
	2 *a.* The SO_2 gas is given off only after standing for a time or on warming, and yellow S separates :— *Presence of a theiosulphate.*		

337. Add strong H_2SO_4 in small quantity to a fresh portion of the of the results enumerated below occurs*, if none of them is noticed

1. *Fumes are evolved with pungent smell and acid reaction to litmus-paper, the liquid is not coloured* :— *Presence of a chloride or nitrate.*		2. *The liquid becomes yellow or red*, and a *chlorous smell is given off:* on warming the tube a crackling noise or slight explosion is produced :— *Presence of a chlorate.*
Confirmatory.—Hold in the fumes a glass rod moistened with a solution of $AgNO_3$ acidified with HNO_3: the solution on the rod becomes milky :— *Presence of a chloride.* Confirm by (351).	*Confirmatory.*—Drop into the acid some small pieces of Cu, reddish-brown gas is evolved :— *Presence of a nitrate.*	*Confirmatory.*—To a portion of the cold solution of the substance in water add a few drops of dilute indigo solution, then some H_2SO_3, the blue colour of the indigo is destroyed.

338. After heating the tube containing the strong H_2SO_4 and porcelain dish for (338*a*); rinse out the tube, and dry the inside *fluoride* is shown. The presence of a fluoride, if decisively indicated

338*a*. Add alcohol to the liquid in the porcelain dish, mix by tinge it shows :—*Presence of a borate.* Confirm by (353).

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* If Pb, Ba, Sr, or Ca is present the addition of H_2SO_4 causes a precipitate of the

† It is best to compare this smell with that evolved when $Na\bar{A}$ is similarly treated.

TION FOR THE ACID-RADICLE.

and observe the effect produced*; then heat to boiling :—
smell; see below :—

5. ***Reddish brown fumes are evolved***, best seen by looking down the tube at white paper. On adding a little *cold dilute* H_2SO_4 and $FeSO_4$ solution to a fresh portion, a deep-brown liquid is produced:— *Presence of a nitrite.*	6. *Smell of HCy.* Test for a cyanide and a ferro-, ferri-, and sulphocyanide in the original substance by (351). 7. *Smell of vinegar.* Refer to (337, 4) below	*No gas is evolved* :— Absence of carbonate, sulphite, theiosulphate, sulphide, hypochlorite, nitrite. Pass on to (337).

substance, mix well by shaking, heat gently and note whether any
proceed to (338) and (338 *a*) :—

3. *A reddish-brown* or *violet* colour is produced in the acid, and no explosion or crackling occurs on warming :— *Presence of a bromide or iodide.* If on heating the mixture, coloured vapour is evolved (see note), hold in the vapour the end of a glass rod covered with moist starch powder: the starch becomes brown :— *Presence of a bromide.* The starch becomes bluish-black :— *Presence of an iodide.* *Note.*—The vapour is much more easily obtained by mixing the substance with MnO_2 powder, before adding H_2SO_4 and heating.	4. *A smell of vinegar* is noticed :— *Presence of an acetate.* *Confirmatory.* — Add to a fresh portion of the substance some alcohol, then strong H_2SO_4; a fragrant smell proves an acetate. †	5. *A mixture of CO and CO_2 gases is given off*: found by a drop of lime-water held in the tube being turned milky, and by the CO burning with a blue flame when kindled :— *Presence of an oxalate.*	6. *The substance blackens* and evolves gases, amongst which SO_2 is recognised by its smell and its action on $K_2Cr_2O_7$ solution :— *Probable presence of a tartrate.* Confirm by (354).

substance, let it stand for some time, then pour the liquid into a
thoroughly; if it is seen to be corroded and dimmed :—*Presence of a*
here, requires no confirmation.
stirring, heat the dish and kindle the alcohol, if the flame has a greenish

---

insoluble sulphate : this may be disregarded.

since a mixture of alcohol and H_2SO_4 when heated evolves a somewhat fragrant smell.

339. GENERAL TABLE FOR THE

If the metal has not been detected in the Preliminary Examination, (see Table pp. 178, 179), and then trying special tests for each

A part of the solution (332) of the substance, *if acid* (340), is mixed with dilute HCl (341);		
The precipitate may be — AgCl—white. Hg_2Cl_2— „ $PbCl_2$— „ Examine for the metal by Table I. (344).	Into the same part of the solution (342) H_2S is passed, or H_2S- is heated to boiling :—	
	The precipitate may be— HgS—black. PbS— „ Bi_2S_3— „ CuS— „ CdS—yellow. SnS—brown. SnS_2—yellow. Sb_2S_3—orange. As_2S_3—yellow. Examine for the metal by Table II. (345).	To a *fresh portion* of the solu- [*Caution.*—If a tartrate or evaporating the liquid must dilute HCl, and tested by If AmHO added after AmCl $AmHMoO_4$ and warmed. *chromate* (343) or fluoride
		The precipitate may be— Al_2Ho_6—{ Colourless and transparent. Cr_2Ho_6—pale-green. Fe_2Ho_6—reddish-brown. $FeHo_2$—{ dingy - green, turning brown in the air. Examine for the metal by Table III.A (346). If the precipitate is opaque or white and has none of the above appearances, refer to (357).

If no metal can be found, the sub-

Notes to the General Table.

340. If the liquid is *neutral* or *alkaline*, a portion of it is made just acid by slowly dropping in dilute HNO_3; if this produces no precipitate or a precipitate which dissolves on adding more HNO_3, another portion of the liquid is examined by the above Table.

If a precipitate forms, which is not dissolved by adding more HNO_3, it may consist of colourless gelatinous H_4SiO_4 from a silicate, or finely divided S which is precipitated *white* from a polysulphide, and *yellow* from a theiosulphate; or of SnO_2, or metastannic acid, or Sb_2O_5 (all white); it may also possibly be SnS_2, As_2S_3 (both yellow), Sb_2S_3 (orange-red), these sulphides being precipitated from a sulphostannate, sulpharsenite, or sulphantimonite.

If H_4SiO_4 separates, it is best to evaporate the acidified solution quite to dryness and then heat the residue with dilute HCl; an insoluble

EXAMINATION FOR THE METAL.

it is most rapidly found by ascertaining to which Group it belongs
member of the Group.

if any precipitate forms, more HCl is added and the liquid heated :—

water is added to it (343), the liquid is then diluted, and if no precipitate has formed it

tion AmCl is added, then AmHO in excess :—
oxalate has been found by (337), some of the solid substance or the residue obtained by
be strongly heated on a piece of porcelain, the residue on cooling is then dissolved in
AmCl and excess of AmHO.]
produces a precipitate, a few drops of the original solution are added to some
Should a yellow precipitate form, indicating the presence of a *phosphate*, or should a
(338) have been found refer to (357) for the examination of the Group IIIA. precipitate.

To the same portion add a few drops of Am_2S, or H_2S-water :—

[*Note*—Before adding Am_2S the liquid may be *blue*, showing *presence of Ni*, or *violet* from dissolved Cr_2Ho_6; the latter must be precipitated by long boiling in a porcelain dish before adding Am_2S.]

The precipitate may be— ZnS—white. MnS—pink. NiS—black. CoS— ,, [FeS— ,,] Examine for the metal by Table III.B. (347).	To the same portion add Am_2CO_3 :—		
	The precipitate may be :— $BaCO_3$—white. $SrCO_3$— ,, $CaCO_3$— ,, Examine for the metal by Table IV. (348).	To the same portion add Na_2HPO_4, warm gently and shake well :—	
		A white crystalline precipitate proves *presence of Mg*.	The metal still present may be NH_4, Na, K; it should have been detected already in the Preliminary Examination by Tests I. or II. (333).

stance is probably an acid (334, II.).

residue confirms the presence of a silicate, it is filtered off and the filtrate tested for the metal by the above General Table.

Any other substance insoluble in dilute HNO_3 is filtered off and may generally be known by its appearance: further, SnO_2 becomes *yellow* and Sb_2O_5 *orange* when moistened with H_2S water, metastannic acid becomes golden-yellow if moistened with $SnCl_2$ solution.

341. Make a note of any changes which occur on the addition of HCl; gases may be evolved (336), or a *yellow* solution may become *reddish-yellow*, which renders probable the presence of a neutral chromate.

342. Before H_2S is added, the liquid must be heated, and if any smell of Cl or of SO_2 or brown nitrous fumes should be perceived, the solution must be boiled down nearly to dryness, then dilute HCl added and H_2S added at once. During evaporation gelatinous H_4SiO_4 may separate (340).

343. The following changes occurring on the gradual addition of H_2S are of importance. If the precipitate is *white* at first, then *brown*, and at last *black*, the presence of Hg″ is shown ; if the solution is strongly acid and gives a *red* precipitate becoming *black* on dilution with H_2S-water, Pb is present.

A separation of white S may occur accompanied by the following changes of colour, which are characteristic and should be noted down :—

A *reddish yellow*	solution changing	to *pale green*	indicates a	ferric salt.
A *reddish yellow*	„	„ *bright green*	„	chromate.
A *green*	„	„ *colourless*	„	manganate.
A *purple*	„	„ *colourless*	„	permanganate.

The precipitate of S is known by being perfectly white and running through a filter, it need not be further examined.

A yellow precipitate which forms only on boiling the liquid may be As_2S_3 from an arsenate, or SnS_2 from a stannic salt.

344. TABLE I.—SILVER GROUP.

A white precipitate forms on addition of HCl, and is either not dissolved when boiled with HCl, or it dissolves and reappears in crystals on cooling the liquid, showing the *presence of Pb.* The precipitate may consist of AgCl, $PbCl_2$, Hg_2Cl_2.

Let the precipitate settle and decant the liquid : boil the precipitate in the test-tube with AmHO :—

1. *The precipitate dissolves*, at least in part, see note below :—	2. The precipitate does not dissolve:—	
Presence of Ag. Confirm by adding excess of HNO_3 to the AmHO solution, a milkiness appears. *Note.*—Coagulated AgCl often dissolves only slowly in AmHO.	*The colour of the precipitate is unchanged:—* *Presence of Pb.* Confirm by decanting the liquid, dissolving the precipitate in $H\bar{A}$, and adding K_2CrO_4 to the solution; a yellow precipitate forms.	*The precipitate becomes black:—* *Presence of Hg′ (Mercurosum).* Confirm by immersing a bright slip of copper in a portion of the original solution, made acid with a few drops of HNO_3; a grey deposit forms on the Cu, which becomes white and bright when rubbed.

345. TABLE II.—COPPER AND ARSENIC GROUPS.

Note the colour of the precipitate yielded by H_2S :—

<table>
<tr><td colspan="4">1. <i>The precipitate is dark brown or black</i> ; it may consist of SnS, CuS, HgS, Bi_2S_3, PbS.
Add pure NaHO in excess to a small quantity of the liquid and precipitate, or of the precipitate alone after decanting the liquid if possible, and boil :—</td></tr>
<tr><td rowspan="2"><i>The precipitate dissolves:</i> to a portion of the NaHO solution add HCl in excess, brown SnS is reprecipitated.
Boil another portion of the precipitate after adding a little yellow Am_2S, then add HCl in excess, a yellow precipitate of SnS_2:—
<i>Presence of Sn''</i> (Stannosum).
The presence of Sn'' should be confirmed by adding $HgCl_2$ to a part of the original HCl solution, a white precipitate shows <i>Presence of Sn''</i>.</td><td colspan="3"><i>The precipitate does not dissolve:</i> it may consist of CuS, HgS, Bi_2S_3, PbS.
<i>a. The original solution is blue or green</i>, and becomes intensely blue on addition of excess of AmHO:—<i>Presence of Cu</i>. Confirm by adding a few drops of H_2SO_4 to the original solution, and dipping into it a bright knife blade or piece of steel, a red film of Cu deposits.
<i>β. The original solution is colourless.</i> Test in separate portions for Hg, Bi, Pb by the following special tests:—</td></tr>
<tr><td>Immerse in the acid solution a bright strip of Cu; a grey film is slowly deposited, which becomes brilliant white when rubbed:—
<i>Presence of Hg''</i> (Mercuricum.)
<i>Caution.</i>—A salt of Hg' may have been converted into an Hg'' salt during solution in HNO_3. The original substance should therefore be treated with AmHO; if it blackens Hg' was present, if not Hg'' was originally in the substance.</td><td>Pour another portion of the original solution into much cold distilled water; a white precipitate or milkiness:—
<i>Presence of Bi</i>.
<i>Note.</i>—If Bi is suspected and no milkiness appears, add excess of AmHO to the liquid; Bi if present falls as hydrate; filter, pour one or two drops of boiling HCl upon the precipitate, and let them drop through into large excess of water; a milkiness on stirring shows <i>presence of Bi</i>.</td><td>To a portion add H_2SO_4 a white precipitate:—
<i>Presence of Pb</i>.
The presence of Pb should be confirmed by adding to a part of the original solution AmHO in excess, then $\overline{HA}$ in excess, then K_2CrO_4: a yellow precipitate shows <i>presence of Pb</i>.</td></tr>
</table>

<table>
<tr><td colspan="3">2. <i>The precipitate is yellow:</i> it may consist of CdS, As_2S_3, SnS_2.
Boil the precipitate with NaHO in excess, after decanting the liquid if possible :—</td></tr>
<tr><td rowspan="2"><i>The precipitate does not dissolve</i> : it is bright yellow and pulverulent—
<i>Presence of Cd</i>.
The presence of Cd may be confirmed by showing that the precipitate produced by H_2S dissolves on being boiled with dilute H_2SO_4.</td><td colspan="2"><i>The precipitate dissolves</i> ; it may consist of As_2S_3 or SnS_2 ; examine portions of the original solution as directed below :—</td></tr>
<tr><td>Acidify a portion with HCl, and place in it a piece of Zn which is known to be free from Sn: Sn is deposited on the Zn :—
<i>Presence of Sn^{iv}</i> (Stannicum).
The deposit of Sn should be detached from the Zn, and dissolved by boiling it in a test-tube with a little strong HCl, then $HgCl_2$ added; a white precipitate confirms the <i>presence of Sn^{iv}</i>.</td><td>Acidify a portion with strong HCl, immerse in it some pieces of bright Cu and boil, a black film is deposited on the Cu :—
<i>Presence of As</i>.
<i>Note.</i>—If the yellow precipitate formed by H_2S appeared at once in the cold, the As is present as <i>Arsenosum</i> ; if it formed only on boiling, <i>Arsenicum</i> is present.</td></tr>
</table>

3. *The precipitate is orange red* : it may consist of Sb_2S_3.

Confirm by acidifying a portion of the original solution with HCl, and immersing in it a piece of Zn resting on a slip of platinum foil, a black stain on the platinum (193) :—
Presence of Sb.

ote.—It occasionally happens, if the solution has not been sufficiently diluted, that H_2S s a reddish precipitate with Pb-solution ; such a precipitate, however, becomes *black* on ing more H_2S-water.

346. TABLE III.A.—IRON GROUP.

The colour of the precipitate produced by addition of AmCl and AmHO will usually indicate whether it contains Fe, Al, Cr; the following tests may be made in confirmation.

To a portion of the original solution add KHO gradually until a precipitate has formed (see Note below Table), then add KHO in excess and stir well, one of the following results will be obtained :—

1. A *colourless* gelatinous precipitate is produced which dissolves in excess of KHO to a *colourless* solution: the precipitate reappears on the addition of much AmCl to the KHO solution :— *Presence of Al.*	2. A *pale green* gelatinous precipitate forms, which dissolves in excess of KHO to a *green* solution; when PbO_2 is added to this green solution and the liquid is boiled and then acidified with $H\bar{A}$, a yellow precipitate is produced :— *Presence of Cr.*	3. A *reddish brown* precipitate forms, insoluble in excess of KHO. Add KCyS to the original HCl solution, a blood-red coloration :— *Presence of* Fe'''.	4. A *dingy green* precipitate forms, insoluble in excess of KHO, the precipitate if filtered off and exposed to the air gradually becomes brown :— *Presence of* Fe''.

Note.—if a colourless gelatinous precipitate has been produced by AmCl and AmHO, and is not produced by addition of KHO, a silicate is probably present. Its presence is confirmed by fusing the solid substance in a bead of $NaAmHPO_4$ (294); or by evaporating the acid solution to dryness, a residue insoluble in acid is SiO_2 and may be tested by the bead as directed above.

347. TABLE III.B.—ZINC GROUP.

The colour of the precipitate produced by addition of Am_2S or of H_2S to the alkaline solution will distinguish Zn or Mn from Ni, Co, and Fe, and will usually distinguish Zn and Mn also from one another. See *a* and *b* below :—

a. If the precipitate is light in colour, add to some of the original solution KHO gradually until a precipitate forms, then add KHO in excess. A *white* precipitate, dissolving in excess of KHO and reprecipitated by addition of H_2S proves *presence of Zn.* A *white or brownish* precipitate, insoluble in excess of KHO and gradually turning dark-brown when filtered off and exposed to the air shows *presence of Mn.*

b. If the precipitate is black it will consist of either CoS or NiS or possibly of FeS; the distinction of the metals Ni and Co by the colour of their solutions—Ni being *green*, Co *pink*—and by the colours they impart to the borax bead (333 IV.) is quite simple. FeS is only precipitated here from a *dilute* solution of a ferrous salt: Fe″ is best detected by (346).

348. TABLE IV.—BARIUM GROUP.

Dip a loop of platinum wire into the original solution or into the HCl solution of the precipitate produced by Am_2CO_3, and hold the wire in the Bunsen flame. If the flame coloration thus obtained is not decisive, employ the tests placed at the foot of each column as confirmatory :—

A yellowish green flame:– *Presence of Ba.*	*A crimson flame*, appearing deep red through the indigo-prism :— *Presence of Sr.*	*A red flame*, appearing dingy green through the indigo-prism :— *Presence of Ca.*
Confirmatory.–Add $CaSO_4$ to a perfectly cold portion of the HCl solution, a precipitate forms *immediately.*	*Confirmatory.*–Add $CaSO_4$ to a perfectly cold portion of the HCl solution, no precipitate is produced, but a precipitate appears immediately on boiling.	*Confirmatory.*–Add $CaSO_4$ to part of the HCl solution and boil; no precipitate. To the rest add AmHO in excess, and $Am_2C_2O_4$: a white precipitate forms.
Note. — A green flame coloration may be produced by H_3BO_3.		

EXAMINATION FOR THE ACID-RADICLE.

349. If the acid-radicle has not been found already in the Preliminary Examinations (335–338*a*), or during the Examination for the Metal (341, 343) or elsewhere, it must be tested for as directed below. Since the solubility of the substance (332), and the metal it contains are known, much trouble is usually saved by referring to the "Table of Solubilities" (440).

For the tests given below, use separate portions of the original substance: try the tests in succession until an acid-radicle is found.

350. Acidify a portion of the liquid, or if a solid dissolve it, with dilute HCl and add $BaCl_2$. If Pb, Hg′, or Ag has been found, acidify with HNO_3 and add $Ba(NO_3)_2$, since HCl and chlorides would precipitate the above metals as chlorides. A white precipitate forms which does not disappear on boiling :—*Presence of a sulphate.*

351. Acidify a portion of the liquid, or if a solid is being examined dissolve the solid, with dilute HNO_3; filter off any precipitate or residue which may form, and add $AgNO_3$ solution : a precipitate forms, which does not disappear on warming. Note the colour of the precipitate ; and according as it is *white*, *yellow*, or *orange*, examine it by 1, 2, or 3:—

1. *The precipitate is white;* it may consist of AgCl, AgCy, Ag_4FeCy_6, or AgCyS. Make a portion of the original solution just acid with HCl, then add Fe_2Cl_6 :—

A deep blue precipitate: *Presence of a ferrocyanide.*	A blood-red coloration: *Presence of a sulphocyanide.*

If Fe_2Cl_6 has yielded neither a precipitate nor coloration, indicating absence of a ferro- and sulpho-cyanide, decant the liquid from the precipitate caused by $AgNO_3$, pour upon the precipitate some strong HNO_3 and boil:

The precipitate remains undissolved: *Presence of a chloride.*	The precipitate dissolves: *Presence of a cyanide.* Confirm the presence of a cyanide in a portion of the original solution by boiling it with $FeSO_4$, Fe_2Cl_6, and KHO, and acidifying with HCl: a blue precipitate: *Presence of a cyanide.*

2. *The precipitate is yellow*, and may consist of AgBr or AgI.
Test some of the original solution for an iodide by adding to it starch solution and KNO_2 solution, then acidifying (if not already acid) with HCl: a deep blue coloration:

Presence of an iodide.

If an iodide is not thus detected, add to another portion of the original solution several drops of Cl-water; if the liquid becomes yellow, and on being shaken with CS_2 colours that liquid reddish brown, it shows:

Presence of a bromide.

3. *The precipitate is orange red*, and is probably $Ag_6Fe_2Cy_{12}$.
Make a portion of the original solution just acid with HCl, then add $FeSO_4$ solution; a deep blue precipitate shows:

Presence of a ferricyanide.

Note.—If Hg has been found, $HgCy_2$ may be present, notwithstanding the formation of no precipitate with $AgNO_3$. If not already detected in (333 I.), refer to (306).

352. Acidify some of the original liquid, or if it is a solid dissolve it, with dilute HNO_3; filter if necessary, add a small quantity of this acid solution to some $AmHMoO_4$ solution, stir well and warm gently:—

A yellow precipitate shows:— *Presence of a phosphate.*	*If no precipitate forms* heat to boiling, the formation of a yellow precipitate shows:— *Presence of an arsenate.*

The presence of a phosphate or arsenate may be confirmed by dissolving the yellow precipitate, separated by filtration or decantation, by pouring upon it a little AmHO; on adding to this solution AmCl and $MgSO_4$ a crystalline precipitate will form, which is to be filtered off and washed with a little cold water: several drops of $AgNO_3$ solution are poured upon this precipitate on the filter; if it becomes *yellow* a phosphate is present, if *brown* an arsenate is present. The presence of an arsenate is further confirmed by a yellow precipitate (As_2S_3) appearing with H_2S when the liquid is boiled.

353. Acidify a portion of the original liquid, or if a solid stir it, with HCl; dip into this solution a piece of turmeric-paper, and dry it at a gentle heat; if the paper becomes *reddish brown* when dry, and changes to *dingy green* or *blue black* when moistened with AmHO, it proves the *Presence of a borate.*

354. The acid-radicles, *one* of which may still be present, are $''\overline{T}$, $''C_2O_4$ and $'F$. If the metal found belongs to Groups I., II., III., or IV., the examination for one of these radicles must be begun at (356), if the metal present is K, Na or NH_4 examine by (355).

355. Make the solution just alkaline with AmHO, then add $CaCl_2$ solution in some quantity, shake well and let stand if no precipitate forms at once: the preliminary examination (337) will usually have indicated which of these acid-radicles is present, and thus show according to which of the three columns below the precipitate is to be further tested :—

I. *A crystalline precipitate, appearing after some time,* shows probable *presence of a tartrate.*	II. *A white pulverulent precipitate* shows probable *presence of an oxalate.*	III. *A gelatinous flocculent precipitate* shows probable *presence of a fluoride.*
Confirm by 1 or 2 below :— 1. Decant the liquid from the precipitate, add a little water and a single drop of AmHO, drop in a small crystal of $AgNO_3$ and warm very carefully; a mirror is formed below the crystal, *presence of a tartrate.* This test may be tried also with the original solution by (322). 2. On acidifying some of the neutral original solution with $H\bar{A}$ and adding alcohol and $K\bar{A}$ (unless K or NH_4 has been detected in the substance), and stirring or shaking well, the formation of a crystalline precipitate shows *presence of a tartrate.*	The presence of an oxalate, already indicated by the test in (337, 5), must be confirmed either by warming some of the original substance with MnO_2 and H_2SO_4, and testing for CO_2 gas (319): or by the more tedious method of filtering off and drying some of the above precipitate of CaC_2O_4, then igniting it and testing for a carbonate with HCl (318 *a*).	Confirm by filtering and warming some of the precipitate, or of the substance if solid, in a small cup of lead or platinum with strong H_2SO_4; fumes will be evolved which etch a properly prepared watch glass (296).

356. An oxalate or fluoride of Ba, Sr, Ca, or Mg will have been already detected. A tartrate of any one of these metals is found by gently heating the finely powdered substance if it is solid, or the neutral solution if a liquid, with *very dilute* AmHO and a crystal of $AgNO_3$: the formation of a mirror shows *presence of a tartrate.* If a metal of Groups I., II., or III. has been found, the metal must be separated before testing for the acid-radicle.

If the metal belongs to Group I. or II., make the original solution just acid with HCl, and add H_2S-solution or pass H_2S into the liquid for about five minutes; filter off the precipitate and add more H_2S to the filtrate, if no more precipitate forms*, boil in a porcelain dish until the liquid ceases to smell of H_2S and examine by (355).

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* If any further precipitate forms add more of the reagent, filter, and test again by adding more of the reagent; this addition of reagent, filtration, and testing the filtrate is repeated until no further precipitate forms on addition of the reagent to the filtrate.

If the metal belongs to Group III. add AmCl, AmHO till alkaline, then Am_2S: boil and filter, add to the filtrate Am_2S, if no more precipitate forms (see footnote, page 198) add $H\bar{A}$ in excess, boil, filter off S if necessary, and examine by (355).

If no acid-radicle can be found in any quantity and the substance is not metallic in appearance, it is probably an oxide or a sulphide; most sulphides yield a sulphate or sulphuric acid when they are boiled with strong HNO_3, which may be detected after dilution by (350); an oxide may often be known by its colour (331) or by some other special property or test, *e.g.* MnO_2 by evolving Cl when it is heated with HCl, and Pb_3O_4 by leaving brown PbO_2 when it is treated with dilute HNO_3.

EXAMINATION OF GROUP III.A.

357. The precipitate formed on the addition of AmCl and AmHO to the solution of a substance insoluble in water may consist of $Al_2H_6O_6$, $Fe_2H_6O_6$, $Cr_2H_6O_6$; or of Fe, Al, Cr, Zn, Mn, Ni, Co, Ba, Sr, Ca, Mg as phosphate or oxalate; or of Ba, Sr, Ca as chromate or fluoride.

The systematic examination of a precipitate in Group III.A. for these substances is a difficult matter; such an examination is however only necessary, when an oxalate (337. 5), a chromate (343) or a fluoride (338) has been already detected, or when a phosphate is found in (339). If a precipitate forms in Group III.A. and the above acid-radicles are absent, proceed as is directed in (346): if the above acid-radicles are present, the metal must be tested for as directed below, unless it has been certainly detected during the Preliminary Examination.

358. Note the appearance of the precipitate caused by AmCl and AmHO, then add Am_2S to the liquid and precipitate, stir well and according to the appearance of the precipitate proceed to examine further by 1 or 2 (page 200).

1. *The precipitate after addition of Am_2S is black*, it may consist of FeS, CoS, or NiS; the means of distinguishing these three metals is given below. Note first the colour of the original solution (334, 1).

a. The colour of the original solution was yellow or reddish-brown:— *Presence of Fe'''.* (*Ferricum.*) This is confirmed by the precipitate produced by AmCl and AmHO in the original solution having been *pale-yellow or reddish-brown*, and becoming black on addition of Am_2S. As a confirmatory test add to a portion of the original solution, acidified with HCl if necessary, a few drops of KCyS solution, a *blood-red* coloration is produced.	*b. The colour of the original solution was pink*, or possibly blue becoming pink on dilution: *Presence of Co.* The presence of Co may be confirmed by adding excess of KHO to the original solution, a blue precipitate turning red on boiling shows *the presence of Co.* Co will also have been detected by its borax bead (333, IV.)	*c. The colour of the original solution was green:—Presence of Ni or Fe''.* Note the effect which was produced by the addition of AmCl and AmHO:—	
		A blue solution was produced:—Presence of Ni. The presence of Ni may be confirmed by adding excess of KHO to a part of the original solution, a light green precipitate shows *the presence of Ni.* Ni will also have been detected by its borax bead (333, IV.)	*A dingy green precipitate*, which, when shaken round in a porcelain dish and left for several minutes exposed to the air, becomes brown:—*Presence of Fe''.* (Ferrosum.) Confirm by adding K_6Cfy_2 to some of the original solution, acidified with HCl if necessary; a dark blue precipitate is produced.

2. *The precipitate, after addition of Am_2S, is white or light coloured.*—It may consist of Cr_2Ho_6, Al_2Ho_6, ZnS, MnS; [or of $BaCrO_4$, or Ba, Sr, Ca, Mg, as phosphate, oxalate, fluoride, silicate, or possibly borate]. The substances in brackets can however only be present if the liquid given for analysis was acid in reaction, or if a solid was given which was insoluble in water.

To a small portion of the original solution add KHO drop by drop until a precipitate is produced. (See Note below the Table.) Note the colour and appearance of this precipitate, then add a larger quantity of KHO and stir or shake well; one of the following results (*a* or *b*) will be noticed:—

a. The precipitate at first produced by KHO re-dissolves when the KHO is added in larger quantity; indicating the presence of **Al**, **Zn**, or **Cr**. Note the appearance of the precipitate:—

A pale green flocculent precipitate:— *Presence of Cr.* Confirm by boiling some of the green KHO solution with lead-peroxide (PbO_2), a yellow liquid is obtained, which, if decanted, yields a yellow precipitate on addition of $H\bar{A}$ in excess. The colour of the solution and of the borax bead will also have detected Cr. *Nota.*—If the Cr was present in the acid-radicle of a chromate, AmCl and AmHO will have yielded no precipitate; but there will have been a green precipitate on addition of Am_2S and heating.	*A white precipitate, which is re-precipitated from the KHO solution,* by addition of a few drops of H_2S-water but is not re-precipitated by addition of AmCl: *Presence of Zn.* Confirmed by no precipitate having been produced by AmCl and AmHO, but a white precipitate by addition of Am_2S, or of H_2S after AmHO.	*A white flocculent precipitate, not reprecipitated* from the KHO solution by addition of a few drops of H_2S-water or by boiling, but reprecipitated immediately if sufficient AmCl solution is added:— *Presence of Al* (see Note). *Note.*—$AlPO_4$ is also precipitated here, and is soluble in KHO, but differs from Al_2Ho_6 in being reprecipitated from the KHO solution by addition of excess of $H\bar{A}$; if PO_4 has been detected the analysis is finished. $AlPO_4$ is soluble only in acids, hence the original liquid must have been acid or the original solid substance insoluble in water.

Note.—Sometimes a flocculent precipitate is produced on adding AmCl and AmHO to the original solution, and yet no precipitate is produced by KHO; this renders probable the presence of an alkaline silicate. In this case evaporate a portion of the solution to dryness with HCl; warm the residue with HCl: if any insoluble residue is left, the *presence of a silicate* is proved; filter and test the filtrate for K or Na.

b. The precipitate at first produced by KHO does not redissolve on addition of more KHO; the precipitate will have one of the following appearances:—

A white or dingy yellow precipitate, rapidly darkening when shaken in the air:— *Presence of Mn.* *Confirmed* in the preliminary examination by producing a green mass when fused with Na_2CO_3 and KNO_3, and by no precipitate having been produced on addition of AmCl and AmHO, unless the solution was exposed for some time to the air.	*A pale yellow precipitate, not darkening in the air:* this precipitate will also have been produced by AmCl and AmHO and its colour will not have been altered by addition of Am_2S:— *Presence of $BaCrO_4$.* Ba has been already detected by the flame coloration (333, II.), and a chromate by the change of colour of the orange red liquid to green on adding H_2S (343).	*A white precipitate, not darkening in the air:—* Presence of Ba, Sr, Ca, or Mg as phosphate, oxalate, &c. Pass on to (359). *Note.*—This precipitate can only be formed if the original solution was acid.

359. *The precipitate may consist of Ba, Sr, Ca, or Mg as phosphate, oxalate, borate*, fluoride, or silicate.*

Examine for the metal and acid-radicle as directed below, working through the columns from left to right, unless the presence of a phosphate (339), oxalate (333, I. 4, or 337, 5), borate (334, V., or 333, II., or 338 *a*), or fluoride (338), has been already proved, in which case proceed at once to examine according to (360), or by column 1, or 2, or 3 below.

If this precipitate is proved to contain a phosphate, oxalate, borate, fluoride, or silicate, the further examination for an acid-radicle becomes unnecessary.

1. Add AmHO in excess to a portion of the original solution†, filter, dry the precipitate and heat a portion of it to a *dull red heat* for several minutes on platinum foil. Place the foil in a test-tube and pour a few drops of dilute HCl upon it; if the residue dissolves with effervescence, it shows the *presence of an oxalate.* Test this HCl solution for Ba, Sr, Ca, Mg, by (339), using it as the original solution. If the ignited precipitate does not dissolve with effervescence, pass on to column 2.	2. Stir some of the precipitate produced by AmHO†, column 1, on a watch glass, with a little water to which a few drops of HCl have been added, until the precipitate is dissolved. Dip into the liquid a slip of turmeric paper, and dry at steam heat; the slip appears *reddish-brown*, and becomes *dingy-green* when moistened with AmHO :— *Presence of a borate.* Examine for the metal present by (361). If a borate is not found, proceed to column 3.	3. Place another portion of the precipitate produced by AmHO†, column 1, in a leaden cup or platinum crucible, add strong H_2SO_4 and warm gently, covering the vessel with a watch glass, which has been coated with a film of wax and has had characters traced with a sharp point through the wax (296); the glass is etched :— *Presence of a fluoride.* Examine for the metal present by (361). If no fluoride is found, test for a silicate by (361).

360. *Examination of a phosphate.*—Add to a portion of the cold original solution AmHO drop by drop, whilst constantly shaking the liquid, until a slight precipitate is formed which does not disappear on stirring; then add $H\bar{A}$ and $Na\bar{A}$ solution, and drop in Fe_2Cl_6 until the liquid after being well shaken has a brown or red colour; boil and filter immediately. The precipitate is thrown away. To the filtrate AmCl is added, then AmHO until it is just in excess, boil and filter; the liquid or filtrate, which must smell strongly of NH_3, is then tested for Ba, Sr, Ca, and Mg by (339), this liquid taking the place of the original solution: and if any precipitate is formed by Am_2CO_3, it must be well washed several times with boiling water to remove Na, since the intense yellow coloration of Na would mask that of Ba, Sr, or Ca.

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*Borates of Ba, Sr, and Ca, are rarely precipitated in Group III., since they are soluble in the AmCl formed on adding AmHO to the HCl solution.

† If a solid is being examined, a portion of the original solid is employed for this test, instead of the precipitate produced by AmHO in the HCl solution.

Explanation of the Method for Examination of a Phosphate.

In order to detect Ba, Sr, Ca, or Mg present as phosphates, the PO_4 must be removed from the solution and the metal left as chloride. This is effected by neutralising all HCl and leaving the solution acidified only by free $H\bar{A}$; by addition of Fe_2Cl_6 in slight excess and boiling, all PO_4 is precipitated as $FePO_4$ which though soluble in HCl is perfectly insoluble in $H\bar{A}$. On filtering, $FePO_4$ remains upon the filter, and the filtrate contains the metal (Ba, Sr, Ca, or Mg) as chloride together with a little Fe, but is perfectly free from phosphate.

361. *Examination of a borate, fluoride, or silicate.*—A portion of the original substance is placed in an evaporating basin, which is then nearly filled with dilute HCl, and the liquid is entirely boiled away; the dish is once more filled with acid, and again evaporated to dryness: the residue in the dish is then warmed with dilute HCl, and filtered if any residue is left:—

Solution: the HCl solution is examined for Ba, Sr, Ca, Mg by (339), this solution being treated as is there directed for the original solution.	*Residue:* if any residue remains insoluble in HCl, it proves:— *Presence of a silicate.*

Explanation of the process.—The evaporation to dryness with HCl removes H_3BO_3 and HF, which volatilise, whilst SiO_2 is left insoluble in HCl and therefore remains as an insoluble powder on afterwards warming with HCl; this residue of SiO_2 also remains, however, if HF was present, being caused by the corrosion of the dish; since however HF if present has already been found, no notice will then be taken of the residue.

THE SUBSTANCE POSSESSES METALLIC LUSTRE.

366. This shows the probable absence of an acid-radicle. The only non-metallic elements likely to be present are free carbon as graphite, free iodine, or combined sulphur, since several sulphides are decidedly metallic in appearance*. The presence of the metals Ni, Co, Fe in the free state, and of Fe_3O_4, may be shown by the substance being attracted by a magnet. Pb, or graphite, will be recognised by marking paper when rubbed upon it, as a black lead pencil does: the mark due to Pb disappears if wetted with dilute HNO_3.

Preliminary Examination.

Experiment.	Observation.	Inference.
I. Heat a portion of the substance in a small ignition-tube	1. A black lustrous mirror forms . 2. A yellow sublimate forms, melting, when heated, to brown drops . . 3. Violet vapour is given off, condensing to black scales on the sides of the tube . . . . . . If the substance sublimes entirely as violet vapour	Presence of As. Presence of S. Presence of I. Iodine alone is present.
II. Heat a portion in the inner blowpipe flame in a cavity scooped on a piece of wood charcoal.	1. Smell of garlic . . . . . 2. If an incrustation forms, refer to par. (333, V.) where in the second and third column will be found the appearance of the incrustation and the metal whose presence it indicates. 3. Smell of burning sulphur . . 4. A green flame coloration . .	Presence of As. Presence of S. Presence of Cu.
III. Place a portion of the substance in a piece of hard glass tube open at both ends, and heat it strongly in the Bunsen flame, and afterwards in the blowpipe flame if necessary.	1. A gas is evolved from the upper end which smells of burning sulphur and turns a piece of filter-paper moistened with $K_2Cr_2O_7$ solution green . . . . . . 2. The substance burns completely but slowly away, glowing when strongly heated. This effect is best seen by heating it strongly on Pt-foil by directing the blowpipe flame on the under surface of the foil .	Presence of S. Presence of C.

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* The most common sulphides which possess metallic lustre are *copper pyrites* and *iron pyrites*, which resemble brass in appearance: and *galena*, which resembles lead.

After completing the preliminary examination proceed as is directed in (332).

No acid-radicle need be tested for except S, As and P, which are most easily detected after they have been converted into acids by oxidation: a portion of the finely powdered substance is dissolved in strong HNO_3, and this solution is diluted with water: H_2SO_4 is then tested for by $BaCl_2$ and H_3PO_4 and H_3AsO_4 by $AmHMoO_4$ (352).

THE SUBSTANCE IS INSOLUBLE BOTH IN WATER AND IN ACIDS.

367. It may consist of any one of the following substances; those included in brackets being less likely to occur than the others, since they are soluble when heated with sufficient water or acid. As will be seen below, the colour frequently serves to indicate what substance is present.

1. *White:* $BaSO_4$, $SrSO_4$, ($CaSO_4$), $PbSO_4$, ($PbCl_2$), AgCl*, SiO_2, (Al_2O_3 ignited), SnO_2, Sb_2O_5, Sb_2O_4, CaF_2.
2. *Dark coloured:* ignited Fe_2O_3 (dark brown or black), ignited Cr_2O_3 (dark green or black), $FeCr_2O_4$ and ignited $PbCrO_4$ (dark brown), C (black).
3. *Yellow:* S, AgBr*, AgI*, (PbI_2).

An insoluble silicate may also be present. For a fuller description of these substances see (475). According to the colour of the substance examine it by (368, 369, or 370).

368. *The substance is white.* If sufficient substance is available try the following preliminary tests; if not, proceed at once to (368 *a*). Pour upon a small portion a drop of Am_2S; if it blackens the presence of Pb or Ag is probable, if it becomes yellow or orange red the presence of Sn or Sb is probable; pass on to test III. in the following Table.

If no change of colour is produced by Am_2S, try tests I and II.

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* Darkens quickly in sunlight, and slowly in ordinary daylight.

Experiment.	Observation.	Inference.
I. Moisten a clean loop of platinum wire, immerse it in the powdered substance, and heat the adhering powder for a short time strongly in the *inner* blowpipe flame; moisten the loop with a drop of strong HCl, and hold it in the Bunsen flame	A *yellowish green* flame.	*Presence of Ba.*
	A *crimson* flame, appearing *deep red* through the indigo-prism.	*Presence of Sr.* Test for SO_4 by Exp. II.
	A *reddish* flame, appearing *dusky green* through the indigo-prism.	*Presence of Ca.*
II. Fuse a portion of the substance, mixed with Na_2CO_3 in fine powder, in a cavity scooped upon a piece of wood charcoal, in the inner blowpipe flame *produced from a spirit lamp flame.*	The cooled mass, if detached from the charcoal, placed upon a bright silver coin, and moistened with water, gives, when crushed with the blade of a knife, a black stain.	*Presence of a sulphate.* *Note.*—If SO_4 is not found, test for F by (338 or 296), and further examine the substance by (368 *a*).
III. Mix some of the finely powdered substance with powdered Na_2CO_3 and KCy, on a small cavity made in a piece of wood charcoal, and heat the mixture in the inner blowpipe flame. If metallic globules form, detach one and strike it smartly with the pestle on the inverted mortar. Also take a globule on the point of a penknife and try if it marks paper as black lead does.	White metallic *malleable* globules, which mark paper, and if dissolved in HNO_3 give a white precipitate with H_2SO_4; a yellow incrustation is also formed on the charcoal.	*Presence of Pb* as chloride, sulphate, or chromate. Refer to the *Note* below this Table.
	White metallic *malleable* globules which do not mark paper; they dissolve in HCl, the solution giving a white precipitate with $HgCl_2$.	*Presence of SnO_2.*
	White metallic *brittle* globules, which, if dissolved in boiling HCl, give an orange red precipitate with H_2S: white incrustation.	*Presence of Sb_2O_4 or Sb_2O_5.*
	White scales, no incrustation: the metal is insoluble in HCl, and if dissolved in HNO_3 gives no precipitate with H_2SO_4 but a white precipitate with HCl; the original substance is instantly blackened by a drop of Am_2S, and is dissolved on being warmed with AmHO.	*Presence of AgCl.* [See also (368 *a*).]

Note.—Boil some of the substance with much water, add HNO_3 and $AgNO_3$, a white precipitate shows *presence of $PbCl_2$.*

Test for chromate by fusing in a porcelain crucible with Na_2CO_3+ K_2CO_3; a yellow mass on cooling shows *presence of $PbCrO_4$.*

The presence of $PbSO_4$ may be proved by test II., and by pouring upon the original substance $H_2\overline{T}$ then excess of AmHO, and boiling:

filter if necessary, acidify a portion of the clear solution with HA, and add K_2CrO_4, a yellow precipitate: *Presence of Pb.* Acidify another portion with HCl, and add $BaCl_2$ a white precipitate insoluble on boiling: *Presence of SO_4.*

If none of the substances mentioned in the above Table have been detected, test for Al_2O_3 by heating the substance strongly on charcoal in the outer blowpipe flame after moistening it with $Co(NO_3)_2$ solution, a blue mass shows *presence of Al_2O_3.*

If Al_2O_3 is not found fuse some of the substance in a clear bead of $NaAmHPO_4$, it floats undissolved:—*Presence of SiO_2, or a silicate.* Proceed to (368 *a*).

368 *a*. Fuse a portion of the finely powdered substance, mixed with four or five times as much fusion mixture, for several minutes in the blowpipe flame on a piece of platinum foil, or in a porcelain crucible if Pb or Ag is present; boil the cool mass with water, filter and wash the residue.

Examination of undissolved residue.—Heat the residue with dilute HNO_3; if it does not dissolve, decant the acid and heat the residue with a small quantity of dilute HNO_3 and HCl mixed: if the residue is still undissolved it must be fused again with fresh fusion mixture, and treated as is directed above.

The acid solution is examined for the metal by (339).

Examination of the water solution.—When the metal present has been detected, it will usually be possible to limit the number of acid-radicles which need be tested for by referring to the list in (367). Tests are given below for all acid-radicles likely to occur in insoluble substances: separate portions of the water solution are to be used.

Sulphate: acidify a portion with HCl and add $BaCl_2$: a white precipitate will form.

Chloride: acidify a portion with HNO_3 and add $AgNO_3$, a white precipitate is produced, which is easily soluble in AmHO. If the precipitate produced by $AgNO_3$ has a yellow tinge and is not easily soluble in AmHO, add to a fresh portion of the water solution a little CS_2, then acidified $CaCl_2O$ solution drop by drop, shaking well between each addition, a brown coloration of the CS_2 shows a *bromide*, a violet coloration an *iodide.*

Silicate: make a portion acid with HCl and evaporate to perfect dryness, if on warming with dilute HCl an insoluble residue is left, this proves the presence of silica or a silicate.

Fluoride: acidify with $H\bar{A}$ and add $CaCl_2$, a gelatinous white precipitate forms. Confirm by tests (338 or 296) tried on some of the original substance.

369. *The substance is dark-coloured* (367, 2).

Place some of the substance on a piece of platinum foil and heat the foil strongly on its under surface by the blowpipe flame :—

The substance burns away slowly but completely :—	*The substance does not burn away;* place upon it three or four times as much powdered Na_2CO_3 and KNO_3, and fuse for some time :—
Presence of C.	1. The substance dissolves, forming a yellow mass when cold :—*Presence of* Cr_2O_3. 2. The substance remains undissolved as a dark brown powder, and the mass on cooling is white :—*Presence of* Fe_2O_3*. Confirm by boiling the mass with water, filtering and fusing the brown residue into a borax bead (100).

370. *The substance is yellow* (367, 3).

Heat a portion of it strongly in a small ignition-tube; one of the following results will occur :—

It fuses and sublimes, the sublimate being yellow and melting to reddish brown drops when heated :—	*It fuses but does not sublime.*
Presence of S. *Note.*—If the substance sublimes entirely it consists only of sulphur.	Examine a portion of the substance for Ag and Pb and for Br and I by (368 *a*).

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* $FeCr_2O_4$ shows both reactions 1 and 2, since it contains both Fe and Cr.

ANALYSES OF SIMPLE SALTS, SHOWING HOW TO ENTER RESULTS.

A. *Liquid given for analysis.*

Preliminary Examination for the Metal.

Experiment.	Observation.	Inference.
1. Noted the colour of the liquid.	Blue.	Presence of Cu.
2. Dipped into it a piece of blue litmus-paper.	The paper was turned red, but on addition of Na_2CO_3 no effervescence occurred.	Presence of a salt with acid reaction.
3. Evaporated a few drops slowly upon a watch glass.	Pale blue residue left.	Presence of some dissolved solid.
Ignited the residue strongly.	Blackened without smell of burning.	Absence of organic acid-radicles.
4. Added KHO solution and boiled.	No smell of NH_3.	Absence of NH_4.
5. Dipped a loop of platinum wire into the solution and held it in the flame.	*Bright green* flame; on moistening the wire with strong HCl and again holding it in the flame it gave a blue coloration.	Presence of Cu.
6. Dipped a clear borax bead into a portion of the residue from 3, and fused in the outer and inner blowpipe flame.	In outer flame the bead was *green* whilst hot, *blue* when cold. In inner flame it became colourless.	Presence of Cu.

Preliminary Examination for the Acid-radicle.

Experiment.	Observation.	Inference.
1. Added dilute H_2SO_4 and warmed.	No gas was evolved.	Absence of carbonate, sulphite, sulphide, and nitrite.
2. Added strong H_2SO_4 and warmed.	No gas was evolved And no red fumes on adding Cu clippings.	Absence of chlorate, &c. Absence of nitrate.

Examination for the Metal.

To a portion of the liquid added HCl:—

No precipitate. Absence of Group I.	Added strong H_2S-water:—
	A brownish black precipitate. Presence of Sn″, Hg″, Bi, Pb, or Cu. Allowed the precipitate to settle, poured off as much of the liquid as possible and boiled the precipitate with excess of NaHO, it remained undissolved. Since the original solution was blue, a clean knife blade was dipped into a portion of it acidified with H_2SO_4, red copper was deposited on the steel:— *Presence of Cu.*

Examination for the Acid-radicle.

Added HCl and $BaCl_2$; a white precipitate was produced which did not disappear on boiling:—*Presence of sulphate.*

Found Cu, SO_4.

B. *Liquid given for analysis.*

Preliminary Examination for the Metal.

Experiment.	Observation.	Inference.
1. Noted the colour of the liquid.	Light yellow.	Presence of a neutral chromate.
2. Dipped red litmus-paper into the liquid.	It was turned *faintly* blue.	Probable presence of an alkali-salt.
3. Evaporated a few drops to dryness upon a watch glass.	A yellow residue remained.	Presence of some dissolved solid.
Ignited the residue strongly.	No blackening.	Absence of organic acid-radicle.
4. Boiled a portion with excess of KHO.	No smell of NH_3.	Absence of NH_4.
5. Dipped a loop of platinum wire into the solution, and held it in the Bunsen flame.	A pale violet flame, appearing crimson through the indigo-prism.	Presence of K.
6. Dipped a clear borax-bead into the residue from 3, and fused in the outer and inner blowpipe flames.	Outer flame: *brown*, hot; *green*, cold. Inner flame: *green*, hot and cold. *Brown* colour not reappearing in the outer flame.	Presence of Cr as a chromate.

Examination for the Metal.

Since the liquid was alkaline in reaction, added to a small portion of it a few drops of HNO_3 until it became acid, this produced no precipitate.

To another portion added HCl, the liquid turned from yellow to orange red, indicating presence of a neutral chromate:—

<table>
<tr><td rowspan="4"><i>No precipitate.</i> Absence of Group I.</td><td colspan="4">Then added H_2S-water and warmed :</td></tr>
<tr><td rowspan="3">The liquid became green, and white S was deposited:
<i>Presence of a chromate.</i></td><td colspan="3">To another portion of the original solution added AmCl, then AmHO, no precipitate formed: then added Am_2S and boiled :</td></tr>
<tr><td rowspan="2"><i>No precipitate</i> in the cold, but on boiling a green flocculent precipitate gradually formed, evidently consisting of Cr_2Ho_6 reduced from the chromate.</td><td colspan="2">Added to another portion of the solution AmCl, AmHO, and Am_2CO_3 :</td></tr>
<tr><td><i>No precipitate.</i> Absence of Group IV.</td><td>Added to the same portion Na_2HPO_4 and stirred :
<i>No precipitate. Absence of Mg.</i></td></tr>
</table>

Since K was found by the flame coloration in the preliminary examination, its presence was confirmed by stirring a portion of the solution, to which a few drops of HCl had been added, with $PtCl_4$ on a watch glass; a yellow crystalline precipitate forming on the lines rubbed by the rod:—*Presence of K.*

Found K, CrO_4.

C. *A white crystalline substance given for analysis.*

Boiled a small portion with water; it dissolved completely.

Preliminary Examination for the Metal.

Experiment.	Observation.	Inference.
1. Heated in a small dry test-tube.	The substance gave off red fumes and oxygen gas, which inflamed a glowing splinter of wood. It left a yellow residue.	Presence of a nitrate of a heavy metal. Presence of Pb, Sn, or Bi.
2. Dipped a loop of moistened platinum wire into the powdered substance, moistened with strong HCl and held in the Bunsen flame.	A pale blue flame.	Presence of As, Sb, Pb.
3. Heated a small portion in a cavity on wood charcoal in the inner blowpipe flame.	Deflagration occurred. A white malleable globule remained which easily marked paper. A yellow incrustation.	Presence of a chlorate or nitrate. Presence of Pb.
4. Fused on charcoal in the inner blowpipe flame with Na_2CO_3 and KCy.	Same result as with 3.	Presence of Pb.

Examination for the Metal in Solution.

Boiled a portion of the powdered substance with water, cooled, added HCl to a part of the clear solution, a white precipitate was formed, which disappeared on boiling, but appeared again in the crystalline form on cooling the liquid (*probable presence of Pb*).

Confirmed the presence of Pb by decanting the liquid and boiling the precipitate with AmHO, it remained white and did not dissolve; decanted the ammoniacal liquid, dissolved the precipitate in a little $H\bar{A}$, and added K_2CrO_4, a yellow precipitate:—*Presence of Pb*.

Since a nitrate was found in the preliminary examination, its presence was confirmed by adding strong H_2SO_4 to the aqueous solution of the substance, and pouring solution of $FeSO_4$ carefully upon the cooled liquid in a test-tube; a brown ring formed on the surface of the acid:—*Presence of* NO_3.

Also some of the solution when heated with Cu and strong H_2SO_4, evolved reddish brown fumes:—*Presence of* NO_3.

Found:—Pb, NO_3.

D. *A white powder, emitting no smell, given for analysis.*

Boiled some of the powdered substance with water, it did not dissolve: added a few drops of strong HCl and boiled, the substance dissolved completely (absence of Group I.) without effervescence.

Preliminary Examination for the Metal.

Experiment.	Observation.	Inference.
1. Heated in a small dry test-tube.	No change.	Absence of volatile and fusible substances and of water of crystallisation, &c.
2. Dipped a moistened loop of platinum wire into the powdered substance, moistened the powder with strong HCl and held it in the Bunsen flame.	No flame coloration.	Probable absence of K, Na, Ba, Sr, Ca, Cu, &c.
3. Heated a portion of the powder strongly on charcoal in the inner blowpipe flame.	The substance did not fuse.	Absence of alkali salt.
	A white luminous residue, which when moistened on red litmus-paper showed an alkaline reaction.	Presence of Ba, Sr, Ca, Mg.

Examination of the solution.

To a portion of the HCl solution added H_2S-water; no precipitate:—*Absence of Group II.*

To another portion of the HCl solution added AmCl and AmHO in excess, a white flocculent precipitate: then added Am_2S, the colour of the precipitate was unchanged.

To a few drops of the HCl solution added KHO drop by drop until a white flocculent precipitate formed, then added excess of KHO and boiled, the precipitate did not dissolve, and it did not become discoloured in the air:—*Presence of Ba, Sr, Ca, Mg as phosphate, oxalate, &c.*

Added a drop of the original HCl solution to some $AmHMoO_4$ solution in a test-tube and warmed gently, a yellow precipitate:—*Presence of PO_4.*

To another portion of the *cold* HCl solution added AmHO drop by drop whilst constantly stirring until a precipitate formed which did not redissolve, then added $H\bar{A}$ and $Na\bar{A}$ solution until this precipitate dissolved when the liquid was well shaken. Fe_2Cl_6 was added until the liquid appeared red, it was then boiled and immediately filtered. To the filtrate AmHO was added in excess and the precipitate filtered off, then Am_2CO_3 was added, a white precipitate formed:—*Presence of Ba, Sr, or Ca.*

Filtered and dissolved the precipitate by pouring upon it a few drops of boiling HCl; dipped a loop of platinum wire into the solution, and held it in the Bunsen flame, a red flame appearing dingy green through the indigo prism:—*Presence of Ca.*

A few drops of the solution boiled with $CaSO_4$ gave no precipitate, proving the absence of Ba and Sr; the presence of Ca was therefore confirmed by the formation of a precipitate on adding to the rest of the solution excess of AmHO, and then $Am_2C_2O_4$.

Found Ca, PO_4.

E. *A white odourless powder given for analysis.*

Boiled a portion of the substance with water, it did not dissolve: added HCl and heated again, it dissolved completely (absence of *Group I.*) without effervescence.

Preliminary Examination.

Experiment.	Observation.	Inference.
1. Heated a portion in a small dry test-tube.	The substance blackened slightly; it evolved CO_2 which turned a drop of lime-water milky. The residue in the test-tube effervesced with HCl whereas the original substance did not.	Presence of an organic salt of K, Na, Ba, Sr, Ca, or Mg.
2. Held a portion of the substance on a loop of platinum wire in the Bunsen flame; then moistened with HCl and again held in the flame.	A *red* coloration, appearing *dusky green* through the indigo-prism.	Presence of Ca.
3. Heated on charcoal in inner blowpipe flame.	A white luminous mass remained, which when moistened on red litmus paper turned it blue.	Presence of Ba, Sr, Ca, or Mg.

Examination for the Metal.

Added to a portion of the HCl solution strong H_2S-water.

No precipitate:— Absence of Group II.	To another portion of the HCl solution added AmCl then AmHO in excess, *a white precipitate formed;* added Am_2S, the colour of the precipitate remained unaltered. To another portion of the solution added KHO drop by drop until a precipitate formed, then more KHO and boiled; a white precipitate insoluble in KHO, and not darkening in the air;—*Presence of oxalate, phosphate, &c., of alkaline earths.* Since the presence of an oxalate of an alkali- or alkaline earth-metal was indicated in the preliminary examination, a portion of the original substance was heated on platinum foil, then dissolved in HCl (effervescence) and to the solution excess of AmHO and of Am_2CO_3 was added. The liquid was warmed and put aside till the precipitate subsided, then the liquid was decanted and the precipitate dissolved in a few drops of HCl. A platinum wire dipped into this HCl solution gave a *red* flame coloration, appearing *dusky-green* through the indigo-prism:—*Presence of Ca.* [Confirmed by another portion of this HCl solution giving a white precipitate with excess of AmHO and $Am_2C_2O_4$.]

The presence of C_2O_4 was confirmed by heating some of the original substance with strong H_2SO_4: gases were evolved which rendered milky a drop of lime-water and burnt with a blue flame, showing presence of CO_2 and CO:—*Presence of* C_2O_4.

Found Ca, C_2O_4.

F. *A yellow metallic-looking substance given for analysis.*

Preliminary Examination.

Experiment.	Observation.	Inference.
1. Heated a portion of the substance strongly in a small tube closed at one end.	A yellow sublimate formed which melted into brown drops: a smell of burning S was evolved, and a piece of paper dipped into $K_2Cr_2O_7$ solution when placed in the mouth of the tube became green. A brown residue left.	Presence of S. Probable presence of Fe.
2. Heated strongly on charcoal in the inner blowpipe flame.	Strong smell of burning S. The residue fused into a dark-coloured globule, which when cold was strongly attracted by the magnet.	Presence of S. Probable presence of Fe
3. Heated a fragment strongly in a glass tube open at both ends, and held obliquely in the flame.	A gas was evolved which had a suffocating smell, and which turned $K_2Cr_2O_7$ paper green.	SO_2 from combustion of S

Solution of substance.

Warmed the powdered substance with a mixture of strong HNO_3 and HCl, it dissolved completely; boiled down in an evaporating dish, adding strong HCl several times; when nearly dry, diluted with water.

Examination of the solution for the Metal.

Since the substance dissolved entirely in the presence of HCl, Group I. is absent; to a portion of the solution added H_2S-water:—

The solution became colourless, and white S was precipitated :—*Probable presence of a ferric salt.*	To another part of the solution added AmCl, then excess of AmHO, a brown flocculent precipitate formed : to a fresh part added excess of KHO, brown flocculent precipitate insoluble in KHO, *Presence of* Fe'''. Confirmed the presence of Fe by adding to a fresh part of the original solution KCyS solution, *a blood-red solution : Presence of* Fe'''.

Confirmed the presence of S by adding to a part of the original solution $BaCl_2$, a white precipitate formed which did not disappear on boiling :—*Presence of* SO_4 *derived from oxidation of sulphur.*

Found Fe, S.

G. *A white powder given for analysis.*

Boiled a small portion with water, it did not dissolve.

Boiled another portion with HCl, it did not dissolve: added HNO_3 and heated again, the substance was not dissolved. Hence the substance is insoluble in water and in acids, and since it is white, it can only be $BaSO_4$, $SrSO_4$, ($CaSO_4$), $PbSO_4$, ($PbCl_2$), AgCl, SiO_2, Al_2O_3, SnO_2, Sb_2O_4, or CaF_2.

Poured Am_2S upon some of the substance: its colour was unchanged, *probable absence of Pb, Ag, Sn, Sb.*

Preliminary Examination.

Experiment.	Observation.	Inference.
1. Took some of the powder up with a loop of moist platinum wire, ignited it strongly in the inner blowpipe flame, moistened it with HCl, and held in the Bunsen flame.	A yellowish green coloration.	Presence of Ba.
2. Fused a portion with Na_2CO_3 on charcoal in the inner blowpipe flame, placed the cooled mass on a bright shilling, and moistened it with a drop of water.	A black stain on the coin.	Presence of S.

Examination of the solution.

Fused some of the powder with fusion mixture on platinum foil: boiled the cold mass with water, filtered.

Filtrate: made acid with HCl and added $BaCl_2$, a white precipitate, which did not disappear on boiling the liquid:—*Presence of SO_4.*

The residue on the filter was well washed with boiling water, and boiling dilute HCl poured upon it, it dissolved completely:—

Absence of Group I.	To a portion added strong H_2S-water and heated:		
	No precipitate. Absence of Group II.	To another portion added AmCl, then AmHO in excess, then Am_2S, and boiled:	
		No precipitate. Absence of Group III.	To another portion of the original solution added AmCl, AmHO, and Am_2CO_3:
			A white precipitate: Presence of Ba, Sr, or Ca. A platinum wire loop dipped into the original solution gave a yellowish-green tint to the Bunsen flame: *Presence of Ba.*

Found Ba, SO_4.

SECTION VI.

FULL ANALYTICAL COURSE AND TABLES.

INTRODUCTORY REMARKS.

After having tried the reactions in the fourth Section and learnt how to detect the members of each group occurring singly or together, substances containing any members of any or all of the groups should be analysed. The substance given for analysis should be either a liquid or a solid, since the full analysis of a gaseous mixture is not provided for in the following course.

PRELIMINARY EXAMINATION OF COMPLEX SUBSTANCES.

374. Before proceding to employ the full course of analysis for substances of complex composition, it will be well to examine a few substances for metals and acid-radicles by the preliminary tables given in paragraphs (383, 384, 385 *et seq.*) For a detailed account of any of these tests the student must refer back to the description given of it in the fourth Section.

375. A little study of these Tables (388–415), will show that they contain certain principal tests marked thus—"Exp. I."; these stand in the first column: in the second column are placed the results which may be noticed on trying these experiments, a glance through these will forewarn the student of what has to be looked for. The main classification of the observations which may be made is indicated by large-type letters, the subsidiary divisions are marked by small numbers and italics, and the individual results are simply placed in succession in a vertical column.

Occasionally an observation is made which it is advisable to confirm by an additional experiment: this *confirmatory test* is then entered in the first column just below the original observation, and is marked *Confirmatory* to distinguish it from the main experiment; its results are placed beside it in the second column. The student must understand that any of the results entered in the second column may be obtained.

A few salts and mixtures such as those mentioned in (560) should be subjected to a careful preliminary examination by the student, and the *results obtained should be carefully entered, as soon as they have been obtained*, in the tabular form as is shown in (488).

FULL EXAMINATION OF SUBSTANCES IN SOLUTION.

After having become familiar with the preliminary examination of solid substances the student should fully analyse some easy substances containing only a few metals and acid-radicles, beginning at paragraph (385) and following up the preliminary examination by dissolving the substance and making a systematic examination of its solution. In examining the solution for metals the following general explanation should be attended to.

376. *It is necessary first to separate the metals into groups* by adding the group reagents in such order that each precipitates the metals of one group only, leaving in solution the metals of all other groups which may be present.

This is effected by adding the group-reagents as is directed in the General Table (419); the Group Table on pages 178, 179, furnishes a convenient explanation of this scheme.

The first reagent added is HCl, which precipitates only the metals of Group I., and leaves the metals of Groups II., III., IV., and V. in solution; hence if a precipitate is obtained on adding excess of HCl, it is filtered off, and any member or members of Group I. will then be present upon the filter, whilst the members of all other groups remain in the filtrate.

On passing H_2S into the filtrate*, metals of Group II. alone will be precipitated and may be filtered off.

The filtrate*, after removal of H_2S and oxidation of any Fe by boiling it with HNO_3, may be mixed with AmCl and excess of AmHO; the AmHO will precipitate any members of Group III.A, Group III.B and Mg remaining dissolved by the AmCl.

The addition of Am_2S to the filtrate* will then separate Group III.B as sulphides.

Group IV. is precipitated from the filtrate* by the last group reagent Am_2CO_3, and the filtrate* is examined for Group V., the members of which, not being precipitated by any group reagent, may now be present in solution.

377. *Addition of reagents in excess.*—Each group must be *completely* precipitated before adding the next group reagent; hence it is necessary, if any precipitate has been formed by a group reagent, to add that reagent *in excess* before proceeding to add the next one: unless the presence of each reagent in excess is made certain before proceeding, much confusion may be caused.

The general method for ascertaining that a group reagent is present in excess consists in adding a few drops more of it to the liquid, which has been rendered clear by letting the precipitate settle or by the filtration of part of it; if any further precipitate is formed, the whole of the liquid should be well stirred with more of the reagent, and the clear filtrate again tested in the same way; this process must be repeated until no further precipitate is caused.

In certain cases this method of proceeding is rendered unnecessary, the excess of a reagent being detectable by its smell after the solution and reagent have been well mixed, and the air above the liquid has been blown out. This is the case for example with H_2S and with AmHO. In other cases, as for example with yellow Am_2S, the excess is seen by the colour of the filtrate. If, however, an acid or alkaline substance is to be added in excess, the student should always prove the excess by the use of test papers.

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* Or solution, supposing no precipitate to have been obtained, and filtration therefore to have been unnecessary.

378. *Washing group precipitates.*—Before examining each group precipitate it is necessary that it should be washed thoroughly from adhering solution, as is described in Exp. 52 (31); this must also be done in the separations by filtration described in the Group Tables. If the washing has not been satisfactorily carried out many complications may arise.

379. *Evaporation before precipitating the Third Group.*—Certain organic substances hinder or prevent the precipitation of the members of Group III.A. by AmHO: these are usually detected in the preliminary examination, and if they are detected, it is necessary to destroy them by evaporation and ignition before proceeding to precipitate Group III.

Evaporation to dryness and gentle ignition are also necessary to separate SiO_2, which, if it remained in solution, might be mistaken for $Al_2H_6O_6$.

But if organic substances, borates, fluorides, and silicates are known to be absent, it is sufficient to boil the filtrate from Group II. until it no longer smells of H_2S, then add a few drops of strong HNO_3 and continue to boil the liquid for several minutes, in order to completely remove H_2S and to convert ferrous into ferric salts.

The separation of the subgroups III.A. and III.B. in the general Table (419) is never complete: for an account of more exact methods the student is referred to paragraphs (459 *et seq.*).

The method of examining the precipitates in Group III. may also be influenced by the presence of a phosphate in the solution: this is fully explained in (463).

380. It will be seen that the full course of analysis is so arranged that it serves not only to detect what is present in a substance, but also to prove that all else is absent: this is usually the object in view when analysing an unknown substance.

The process may in most cases be shortened by using separate portions of the solution: the plan adopted in the general Table of working throughout with the whole of the solution is however preferable, since it enables the analyst to form an opinion as to the relative quantities of the different substances present; and this is usually important.

An example of entry of results is given in (488); it will be seen that the form adopted is that of the Analytical Tables.

Substances for analysis are mentioned in (560, 561)

The student will do well at first to gain much experience in the analysis of easy mixtures, which are completely soluble in HCl and contain no cyanogen, organic salts, silicates, or phosphates precipitable in the Third Group. The analysis will thus be much simplified, since it will be unnecessary to evaporate the filtrate from the second group to dryness, or to follow any special method of precipitating and examining Group III.

When the ordinary course of analysis has been mastered, substances should be attempted in which the above complications exist: and finally minerals, water residues, and artificial products and bye-products should be analysed, in which minute traces of their constituents are carefully looked for.

It will be unnecessary to do more than mention the extreme importance of the spectroscope in rapidly discovering certain substances, especially when they are present in minute quantity only (42).

ANALYTICAL TABLES.

GENERAL COURSE OF ANALYSIS FOR SOLID AND LIQUID SUBSTANCES.

381. Unless cyanogen is known to be absent, it must be tested for in a small portion of the substance by the tests described in (304, 306). If cyanogen is detected refer to (482 *et seq.*) for the preparation of the solution and the method of analysis. If cyanogen is not present, the substance is examined by (383, 384) or by (385 *et seq.*) according as it is a liquid or a solid.

382. A silicate may also be tested for by fusing some of the finely powdered substance in a bead of microcosmic salt (294). If a liquid is being examined, a portion of it is evaporated to dryness, and the solid residue is then examined.

I. THE SUBSTANCE FOR ANALYSIS IS A LIQUID.

383. Test it with blue litmus paper and with either red litmus or turmeric paper: one of the following results will be obtained (34):—

1. *It is neutral,* not changing the colour of either paper: this shows the absence of acids and alkalis and of salts with acid or alkaline reaction. The salts of Ag and Mg and certain salts of Am, Na, K, Ba, Sr, and Ca are the only neutral soluble salts.

2. *It is acid,* turning *blue* litmus *red:* this proves the presence of an acid, or of a salt with acid reaction.

3. *It is alkaline,* turning *red* litmus *blue,* or turmeric *brown:* indicating the presence of Am, Na, K, Ba, Sr, or Ca as hydroxide, or of a salt with alkaline reaction.

384. Evaporate a few drops of the liquid upon platinum foil, thin glass, or porcelain, smelling it occasionally :—

1. *A residue is left.*—Examine the liquid by the further preliminary tests in (334): or evaporate a portion of the solution to dryness in a porcelain dish, avoiding heating the substance after it is dry, and examine the residue by the preliminary tables for metals and acid-radicles (385–417). Examine the larger part of the solution for metals by the general Table (419) paying attention to (420): reserve the rest of the solution for the examination for acid-radicles by (440 *et seq.*).

2. *No residue is left.*—The liquid must consist of some volatile substance, probably of water; this is either pure or contains certain gases or volatile substances such as $(NH_4)_2CO_3$, CO_2, NH_3, HCl, Br. These may be detected by their smell, by the action of the liquid on litmus paper, or by special tests. If the liquid has no action on litmus, no smell, leaves no residue on evaporation and is tasteless, it is pure water.

II. THE SUBSTANCE FOR ANALYSIS IS A SOLID.

385. Carefully note the appearance of the substance. If it is lustrous and metallic, refer to (467) for the method of analysis; if it is non-metallic in appearance, proceed to further examine the substance as follows.

Note down anything that can be learned about the physical properties of the substance by the use of the senses, aided by a magnetised penknife blade and a lens or microscope.

Note especially whether the substance consists of more or less sparkling particles with similar geometrical shape, when it is said to be *crystalline;* or whether the particles are *amorphous* and irregular in shape.

Also test the *hardness*, by ascertaining whether it is readily powdered; or, if the particles are sufficiently large, by finding the ease with which they are scratched by the point of a penknife; very hard substances, such as quartz, cannot be scratched by a knife.

Try whether it is *magnetic* by immersing the tip of a magnetised blade into the finely powdered substance, and seeing whether the particles are attracted; Fe and certain of its compounds are the most powerfully magnetic bodies known, but Mn, Ni, Co and certain of their compounds are also magnetic.

Note also whether the substance possesses any characteristic *smell* or *colour*. The following are some of the more commonly occurring coloured compounds:—

Blue: hydrated cupric salts, and anhydrous cobalt salts.
Green: certain Fe″ salts are pale green; $CuCl_2$, salts of Ni, manganates, and certain compounds of Cr, are intense green.
Yellow: HgO, normal chromates; Fe‴ salts are brownish yellow.
Red: HgO, HgI_2, HgS, Pb_3O_4; dichromates are orange red.
Pink: salts of Mn are delicate pink; hydrated salts of Co are reddish pink.
Brown: Fe_2O_3, PbO_2; PbO is light brown.
Black: CuO, MnO_2, Sb_2S_3, FeS.
White: anhydrous salts of Cu and Fe″, and many other substances.
Colourless: a large number of colourless substances are known; these, as well as many of the faintly coloured bodies, yield *white* powders when crushed.

386. After completing the above examination, the solid substance is reduced to the finest powder possible by rubbing it in the mortar. An ordinary Wedgwood mortar may be used for powdering substances which are pretty soft; but hard rocks and minerals frequently require to be first crushed in a clean bright steel mortar, by blows of a hammer, and to be afterwards pulverised in an agate mortar.

The subsequent chemical examination of the substance is very greatly facilitated by carefully reducing it to an impalpable powder, which does not feel gritty when rubbed beneath the pestle or between the fingers.

387. A portion of this powder is submitted to the tests described in the following preliminary examinations for metals and acid-radicles: the remainder being reserved for the fuller method of examination by solution (418 *et seq.*).

Experiment.	Observation.	Inference.
388. EXP. I.—Heat a small quantity of the substance in a small test-tube or piece of hard glass tubing drawn off at one end.	A. The substance does not change.	Absence of *organic* substances which blacken and give off a smell of burning; of *volatile* substances which sublime; of substances which are altered by ignition; and of *water* combined with or absorbed by the substance, which would be evolved as steam and form drops on the side of the tube.
	B. The substance changes. 1. *It changes colour*:—	
	Yellow, hot. *white*, cold.	ZnO.
	Yellow, brown " *yellow* "	PbO.
	Yellow-brown " *yellow* "	SnO_2 or Bi_2O_3.
	Dark red or *black* " *red-brown* "	Fe_2O_3.
	The substance blackens— *a*. Emitting a smell of burning. The black carbon may be burnt off by strongly heating on platinum foil with the blowpipe flame.	Presence of organic matter.
	Smell of acetone.	An acetate.
	Smell of burning sugar.	A tartrate.
	b. Emitting no smell of burning. The colour is not removed by ignition as under *a*.	Probable presence of Co- or Cu-salts.
	2. *It fuses, and becomes solid again on cooling.*	Salts of alkalis or certain salts of the alkaline earths.
	3. *It sublimes.*	Compounds of Am, As, Hg; or free S or I.
Confirmatory.—The sublimate is carefully examined, if necessary, with the aid of a lens.	White, crystalline (sparkling) sublimate,	$HgCl_2$, As_2O_3.
	White, non-crystalline sublimate.	Probably Am-salt.
	Black sublimate, becomes red when rubbed.	HgS.
	Yellow sublimate.	As_2S_3. HgI_2, becomes scarlet when rubbed.
	Sublimate of reddish drops, which are yellow when cold.	S free or from certain polysulphides.
	Violet vapour, cooling to blackish crystals.	Presence of I.
389. *Confirmatory*.—Heat another portion of the substance, mixed with about three times as much Na_2CO_3 in fine dry powder and a little KCy, in a tube closed at one *end* (147).	Grey mirror, which, when rubbed with a splinter of wood or a glass rod, or when examined by a lens, is seen to consist of globules of Hg.	Presence of Hg.
	Blackish-brown shining mirror, and smell of garlic; no globules.	Presence of As.

Experiment.	Observation.	Inference.
390. *Confirmatory.*—Pour a little strong KHO solution upon some of the substance and heat to boiling; or mix some of the substance with soda-lime in a mortar, and moisten and heat.	NH_3 is given off, known by its smell and by turning moist red litmus-paper blue or turmeric-paper brown; the change in colour is only a trustworthy indication if it occurs very soon, as the NH_3 in laboratory air often causes it to occur after a time.	Presence of NH_4.
	4. *The substance gives off water.*	Presence of water, absorbed or combined.
	It fuses first, then gives off water, and again becomes solid if the heat is continued.	Water of crystallisation.
	It swells up considerably whilst giving off its water.	Borax and certain borates, alums, and phosphates.
391. *Confirmatory.*—Examine the drops of water on the sides of the tube with blue and red litmus-papers.	The water is alkaline.	Probably NH_4-compounds.
	The water is acid.	Presence of volatile acids, *e.g.*, HNO_3, HCl, H_2SO_4.
	5. *Gas or vapour is given off*.* *a. It is without smell.*	
392. *Confirmatory.* — Introduce a burning splinter of wood into the tube.	It burns more brightly, and if introduced with a spark at the end is inflamed,	O from chlorates, nitrates, peroxides, &c.; or possibly N_2O.
	The flame is extinguished.	CO_2 or N.
393. *Confirmatory.* — Introduce a glass rod, moistened with lime-water, into the tube.	The lime-water turns milky.	CO_2 from carbonates, oxalates, &c.
	The lime-water does not turn milky, but the flame is extinguished (392).	N from $AmNO_2$ probably.
	b. The gas or vapour has a smell.	
	Smell of burning S.	SO_2 from combustion of free S, or from acid sulphites, thiosulphates, reduction of sulphates or oxidation of sulphides.
394. *Confirmatory.* — Introduce a glass rod or slip of paper moistened with $K_2Cr_2O_7$.	The $K_2Cr_2O_7$ turns green; moist blue litmus is also reddened by the gas.	
	Reddish-brown nitrous fumes are given off, known by their peculiar smell, and by not colouring starch paste orange red.	Nitrates of heavy metals (*e.g.* of Pb, Bi, Hg. &c.)
	Smell resembling Cl:—	
395. *Confirmatory.* — Introduce a glass rod carrying a piece of moist litmus.	Yellowish gas, which bleaches moist litmus.	Cl from certain chlorides.
396. *Confirmatory.* — Introduce a glass rod with moist starch powder on its end.	Brown vapour, which colours the moist starch powder orange red.	Br from certain bromides.
	Violet vapour, which colours starch solution blue.	I, free or from certain iodides.
	NH_3 is smelt.	NH_4 or cyanogen compounds.

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* This will usually be shown by effervescence of the substance; if the gas or vapour is not detected by its colour or smell, it can only be found on testing for it specially by (392) and (393).

Experiment.	Observation.	Inference.
	c. The gas can be ignited, and burns at the mouth of the tube with a:—	
	Pale *yellowish green* flame, more or less explosive.	NH_3 probably from strongly heated NH_4NO_3.
	Bright *white* flame, producing white fumes; the unburnt gas smells of garlic.	PH_3 probably from a hypophosphite.
	Peach-blossom coloured flame.	Cy from a cyanide, probably from $HgCy_2$.
397. Exp. II.—Dip a moistened loop of platinum wire, which gives no colour to the Bunsen flame, into the substance, and hold the loop with the adhering powder near the top of the Bunsen flame, or in the inner blowpipe flame. Then moisten with a drop of strong HCl and heat again. Continue the heating until no further change occurs in the colour of the flame. The flame coloration should also be carefully examined by means of the spectroscope (42). *Note.* If silicic acid is present, the colorations for K and Na usually show only after ignition of the powdered substance with powdered $CaSO_4$.	A. The substance colours the flame:	
	1. *Intense yellow.*	Na.
	Examine the flame through the indigo-prism; a crimson colour is seen.	Presence of K or Sr, or both.
	2. *Pale violet,* crimson through the indigo-prism.	K.
	3. *Yellowish green.*	Ba probably.
	4. *Crimson,* same through the indigo-prism.	Sr.
	5. *Orange red,* dingy green through the indigo-prism.	Ca.
	6. *Bright green.*	Cu or B_2O_3.
	A blue colour is seen after moistening with strong HCl.	Cu.
	7. *Blue* { *Intense.*	$CuCl_2$, $CuBr_2$.
	Livid.	As, Sb, Pb.
	B. The substance does not colour the flame.	Probable absence of the above.
398. Exp. III.—Heat the finely powdered substance in a small cavity scooped in a piece of wood-charcoal, in the blowpipe flame.	A. The substance decrepitates, or crackles.	NaCl and certain other crystalline salts.
	B. The substance deflagrates, or causes charcoal to burn rapidly.	Chlorates, nitrates, &c.
	C. The substance fuses easily, and is absorbed by the charcoal, or forms a liquid bead.	Salts of alkalis and certain salts of the alkaline earths.
	D. An infusible residue is left on the charcoal:—	
	1. *The residue is white and very luminous.*	Probably BaO, SrO, CaO, MgO, Al_2O_3, ZnO (or SiO_2)
399. *Confirmatory.*—Detach a portion of the cool residue from the charcoal, place it upon a piece of red litmus-paper, and moisten with a drop of water.	The paper turns blue, showing the residue to be alkaline.	BaO, SrO, CaO, and possibly MgO.

Experiment.	Observation.		Inference.
400. *Confirmatory.*—Moisten the residue on the charcoal when cool with several drops of $Co(NO_3)_2$ solution, and heat again strongly in the outer blowpipe flame.	A *blue* residue, the colour of which does not disappear with intense heat.		Al_2O_3, and some phosphates, silicates, and borates.
	A *pink* residue.		MgO.
	A *green* residue.		ZnO.
401. *Confirmatory.*—Heat a small quantity of the substance in a clear colourless borax bead, first in the outer, then in the inner blowpipe flame.	2. *The residue is coloured.* Colour of bead:		
	In outer flame.	In inner flame.	
	Green, hot, and *blue*, cold.	*Colourless*, or *red*, cold.	Cu.
	Blue, hot and cold.	*Blue*, hot and cold.	Co.
	Violet, hot, and *yellow*, cold.	*Grey* or *opaque*.	Ni.
	Brown-red, hot: *yellow*, cold.	*Olive green*, hot and cold	Fe.
	Green, hot and cold.	*Green*, hot and cold.	Cr. See (402).
	Reddish purple, hot and cold.	*Colourless*, hot and cold.	Mn. See (402).
402. *Confirmatory.*—The presence of Mn and Cr, may be confirmed by fusing the substance with Na_2CO_3 and KNO_3 on platinum foil.	*Light-yellow* mass on cooling.		Cr.
	Blue-green mass on cooling.		Mn. (See 406.)
403. *Confirmatory.*—Heat also some of the substance, mixed or covered with powdered KCy and Na_2CO_3 or $K_2C_2O_4$, on charcoal in the inner blowpipe flame.	3. *The residue is coloured, or metallic scales or globules are seen.* The substance is reduced to the metallic state. (See 407.)		Au, Ag, Cu, Pb, Sn, Sb, Bi; [Co, Ni, Fe, Mn, form grey powders].
	a. Without incrustation.		Au, Ag, Cu, Co, Ni, Fe, Mn.
	Brilliant white metal.		Ag.
	Yellow metal.		Au.
	Red scales or globules.		Cu.
	Grey powder, attracted by a magnetised knife-blade (407).		Fe, Co, Ni, Mn.
	b. With incrustation.		
	Globules.	Incrustation.	
404. *Confirmatory.*—Detach several of the globules with the point of a knife, and strike them on the bottom of an inverted mortar with the pestle; if they flatten to a cake they are *malleable*, if crushed to a powder they are *brittle*. Try also if a globule, fixed on the point of a knife, marks paper.	*White, malleable*, not marking paper.	None, or very slight.	Sn.
	White, brittle.	*Orange*, hot. *Yellow*, cold.	Bi.
	White, malleable, marking paper.	*Yellow*, hot and cold.	Pb.
	White, brittle, giving white fumes.	*White*, close to substance.	Sb.
Note.—The appearance d malleability of one duced metal is fre-ently much altered by presence of another.	*White* or none.	*Yellow*, hot. *White*, cold.	Zn.
	White or none.	*Red-brown*, easily volatilised.	Cd.
	None.	*White*, and white fumes; smell of garlic.	As.

Experiment.	Observation.	Inference.
405. Exp. IV. — Fuse some of the substance, mixed with Na_2CO_3, on charcoal in the inner blowpipe flame *produced from a spirit-lamp;* remove the fused mass when cold, place it on a bright silver coin, moisten with a drop of water, and let stand for several minutes.	On rinsing off the substance, the coin is found to be stained black (228). Also, if a drop of HCl be placed upon the mass, a smell of H_2S is perceived, and lead-paper is blackened.	Presence of S, free or combined. *Note.* — Since this reaction serves to detect S and sulphur-acid-radicles, it more properly belongs to the Preliminary Examination for acid-radicles: it is, however, best tried here.

Notes to the preceding Preliminary Table.

406. The green colour produced by Mn conceals the light yellow due to Cr; on boiling the residue with water it gives a pink or purple solution, the colour of which is best seen on filtering: this colour is due to the formation of $KMnO_4$, and shows Mn to be present. On acidifying this solution with $H\bar{A}$ and boiling again for several minutes, the purple colour is destroyed; and on filtering, the yellow colour due to Cr is seen: the presence of Cr may be further confirmed by the formation of a yellow precipitate in the acid solution on addition of $Pb\bar{A}_2$.

407. The metal is best separated and examined by detaching the mass when cold from the charcoal, and powdering it by crushing in a mortar or on a watch glass with a little water, letting stand for a short time, and then quickly pouring off the water down a glass rod or pestle which is wetted and pressed against its edge; by several times repeating this operation, the heavier metallic particles alone are left in the mortar or watch glass (40).

409. *Note.*—When the substance to be examined contains several bodies, they frequently more or less mask one another's reactions: thus Co if mixed with Fe will give a bead green whilst hot and blue when cold, thus resembling Cu, but differing in remaining blue in the inner flame: hence the composition of many complex mixtures is only roughly indicated by the Preliminary Examination, and must be confirmed and established in the wet way. Many substances, more particularly minerals, however, can be completely analysed by a careful Preliminary Examination.

PRELIMINARY EXAMINATION FOR ACID-RADICLES.

410. The substance is required for this examination in the state of powder or of *strong* solution.

If the substance given for analysis is a solution, part of it may be evaporated to dryness: the dry residue is then finely powdered and subjected to the following tests. If several acid-radicles and metals are present, the reactions may be more or less perfectly concealed or altered, hence failure in obtaining a certain reaction does not in all cases necessarily prove the absence of an acid-radicle.

Experiment.	Observation.	Inference.
411. Exp. I.—Treat some of the substance with dilute HCl and note the result, then heat moderately.	*One or more of the following gases may be evolved:—*	
	A colourless gas without smell, which turns milky a drop of lime water on the end of a glass rod.	CO_2 from a carbonate.
	A gas of suffocating smell, which turns a drop of $K_2Cr_2O_7$ solution green.	SO_2 from a sulphite, or from a sulphate in the presence of As_2O_3 or other reducing substance.
	Yellow S is precipitated at the same time.	SO_2 and S from a thiosulphate.
	A gas with fetid odour, which blackens a drop of $Pb\bar{A}_2$ solution, or a piece of lead-paper (416).	H_2S from a sulphide; or possibly from a sulphite or thiosulphate, if Zn or other reducing agent be present.
	Reddish fumes.	N_2O_3 from a nitrite.
	A yellowish green gas, of suffocating smell, which bleaches moist litmus paper, is evolved:—	
	a. In the cold; and also evolved on adding $H\bar{A}$.	Cl from hypochlorites.
	b. Only when heated.	Cl from action of HCl on oxidising substances, *e.g.*, MnO_2, chromates, nitrates, chlorates, &c.
	A colourless gas, smelling of bitter almonds.	HCy from a cyanide.
Dip into the HCl solution a strip of turmeric paper.	On drying this paper at a gentle heat, it becomes reddish brown.	Presence of a borate (283).

Experiment.	Observation.	Inference.
412. Exp. II. — Heat another portion gently with strong H_2SO_4. No attention need be paid to gases evolved in Exp. I. (411).	1. A gas is evolved with pungent smell, which fumes in the air, and renders milky a drop of $AgNO_3$ solution made acid with HNO_3.	HCl, HBr, HI, HF* from chloride, bromide, iodide, or fluoride.
Confirmatory.—Mix some of the substance with MnO_2 *free from chloride*, add strong H_2SO_4 and warm.	A *yellow* gas is given off with strong smell, which bleaches moist litmus-paper (417).	Probably Cl from a chloride.
	Brown vapour is given off, which colours a little moist starch powder orange red.	Br from a bromide.
	Violet vapour is given off, which colours a drop of starch paste blue.	I from an iodide.
	2. A heavy fuming suffocating gas is evolved, and the liquid behaves as if the glass were greasy; on rinsing out the tube, and drying it thoroughly, the inside is seen to be corroded and dimmed.	HF from a fluoride: confirm by (296). The HF acting upon the silica of the glass evolves SiF_4, which is detected by holding a moistened glass rod in the gas, SiO_2 is deposited as a gelatinous film upon it.
413. *Confirmatory.*—Drop into the hot liquid a few small pieces of copper. If no brown fumes are seen proceed to (414).	3. The acid fumes are reddish, usually seen only on *heating* after adding a few fragments of Cu.	Presence of nitrate. If iodide is present it must usually be separated by Cu_2SO_4 (266) before a nitrate can be detected.
414. *Confirmatory.*—Boil some of the substance with water, or take some of the solution if a liquid; cool, add about twice as much strong H_2SO_4, cool, and pour cold solution of $FeSO_4$ carefully in upon the top of the acid liquid.	A dark brown ring or layer forms upon the surface of the acid, best obtained on cooling the liquid by immersing the test-tube in cold water.	Presence of a nitrate.
	4. The acid is coloured reddish yellow, and a a yellow gas is evolved which smells like Cl and bleaches litmus; on heating the acid explosion or crackling occurs. Confirm by (252.)	Presence of a chlorate. For detection of a nitrate and chlorate when mixed, see (256).
	5. The substance changes from yellow to green, O being evolved as is shown by a glowing taper.	Presence of a chromate

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* HF would render the drop milky by depositing SiO_2 from SiF_4, formed its action on the glass, but it is distinguished by corroding the glass. 412, 2, note.)

Experiment.	Observation.	Inference.
	6. Yellowish green gas, with suffocating smell, which bleaches moist litmus.	Cl from a hypochlorite (detected already in 411), or from a chloride in presence of MnO_2 &c.
	7. The same gas as in 6, but coloured *reddish brown* by nitrous fumes: or coloured reddish brown by $CrOCl_2$.	A chloride in presence of a nitrate or nitrite. A chloride in presence of a chromate.
	8. Substance does not blacken, but evolves CO which burns with a blue flame, and CO_2 which turns a drop of lime-water milky.	Presence of an oxalate.
	9. Substance blackens, and SO_2 is smelt.	Presence of a tartrate.
	10. A smell of acetic acid or vinegar is noticed.	$H\bar{A}$ from an acetate.
415. *Confirmatory.*—Add a little alcohol to the substance and heat with H_2SO_4.	Strong fragrant odour.	Ethyl acetate from an acetate.

NOTES ON THE PRECEDING TABLE.

416. The S present in sulphides which are not decomposed by dilute HCl, is detected by fusing the powdered substance with two or three times as much fusion mixture ($Na_2CO_3 + K_2CO_3$) in a covered crucible, boiling with a little water, and placing a drop upon a bright silver coin, when a dark stain will be produced; or by adding excess of an acid to the solution, when H_2S will be evolved. Smaller quantities of S may be detected by the formation of a black colour or precipitate on adding $Pb\bar{A}_2$ to the solution of the fused mass.

417. Chlorides treated in this manner evolve Cl, recognised by its colour, smell, and bleaching action on litmus-paper; but this test for chlorides is trustworthy only if the precautions stated in (259) are attended to, since many samples of MnO_2 evolve Cl when warmed with H_2SO_4.

EXAMINATION FOR METALS.

The preliminary examination of the solid substance must be followed by preparing a solution of it, and submitting this to the more full and systematic examination, which commences in (419). This is often termed "The Examination in the Wet Way."

PROCESS OF SOLUTION.

418. A portion of the solid, reduced to an impalpable powder (386), is boiled in a flask or boiling tube with distilled water: if it dissolves entirely, proceed to (419).

If the powder does not dissolve, allow the liquid to stand until the undissolved portion has in great part settled, then decant through a filter: the filtrate is solution I.:—

Residue in flask: boil with dilute HCl (418*a*), allow any residue to settle, decant through the filter used above (418*b*); repeat this process and finally boil the residue with strong HCl, let settle, decant through the filter:—			
Filtrate is	*Residue:* heat with dilute, then with strong HNO_3 (418*c*) as was directed above for HCl; decant through the filter:—		
	Filtrate is	*Residue* may be warmed for a short time with a mixture of strong HNO_3 (386*a*) with three times as much HCl; if this does not dissolve it, dilute and decant the acid mixture through the filter:—	
		Filtrate is	*Residue:* wash well with water, dry, and examine as directed (475 *et seq.*) for substances insoluble in water and acids.
Solution II.	Solution III.	Solution IV.	

Solutions I., II., III., IV. need not be examined separately. Add to I. a few drops of dilute HNO_3, and if no precipitate is produced (420) acidify it with HCl, add also to solution III. some HCl: any precipitate caused by HCl in these solutions is filtered off and examined by Table I. The solutions I., II., III., IV. are then mixed together and any precipitate produced by mixing them is examined by (475) as an insoluble substance.

It is best to boil down solutions III. and IV. considerably, in order to get rid of most of the HNO_3 which they contain, before mixing them with I. and II. The solution is then examined by (419).

NOTES ON THE PROCESS OF SOLUTION.

418 *a*. Any changes which occur on the addition of HCl should be noted down. Notice especially whether any gases are given off: many of these may be recognised by their smell, but a closer examination of them may be neglected, as they will have been already obtained in the preliminary examination for acid-radicles (411). If the gases come off from the aqueous solution on adding HCl, or on mixing the HCl and aqueous solutions, they must have been present in salts soluble in water, probably salts of alkali metals; if they are evolved on treating the residue insoluble in water with HCl, they were present as salts insoluble in water.

418 *b*. On cooling the hot HCl solution crystals often separate, these usually consist of $PbCl_2$, rarely of $BaCl_2$: $BaCl_2$ will, however, dissolve easily on the addition of water. The crystals are filtered off and dissolved in a little boiling water; Ba is easily found by trying the flame coloration given by the solution, and if on addition of K_2CrO_4 to the solution a bright yellow precipitate forms, the *presence of Pb* is proved.

If Pb has been found and a residue is left after treatment with HCl, this residue, which may consist of $PbCl_2$, may often be dissolved by boiling it with water, the use of HNO_3 being thus avoided.

418 *c*. HNO_3 seldom requires to be used in dissolving a substance, and it should be employed only in very small quantity, since in the course of analysis it decomposes H_2S with separation of S, unless the solution is dilute and cold (423), and thus hinders the precipitation of Group II.

If a complex solid mixture is given for analysis, and the only object is to obtain a solution as rapidly as possible, the substance may be at once boiled with aqua regia for a few minutes, then diluted and filtered: the residue is examined as a substance insoluble in water and acids (475 *et seq.*): the filtrate is evaporated nearly to dryness, dilute HCl is added, and *the solution is examined* by the General Table (419).

419. GENERAL TABLE FOR SEPARATION OF

These lines run across both pages.

If the substance for analysis is a liquid with *neutral* or *alkaline* reaction, it examined as directed below.

In this Table, and also in the Group Tables which follow, it has been sup-precipitate will be produced when the reagent is added for its detection ; and

<table>
<tr><td colspan="3">Add to the greater part of the "original solution" * HCl; if any precipitate is produced,</td></tr>
<tr><td rowspan="3">The precipitate may contain—
$PbCl_2$—*white.*
AgCl—*white.*
Hg_2Cl_2—*white.*
(See 422.)
Examine the precipitate by Table I. (433).
Note.—When the hot HCl solution is cooled, $PbCl_2$ often separates in white crystals. The absence of Pb is not proved, if it is not precipitated in this group, as $PbCl_2$ is somewhat soluble, and Pb may therefore be found only in Group II.</td><td colspan="2">The filtrate or solution, which must give no further precipi- then diluted with water (425), unless it is already dilute; several minutes (426, 426 *a*) and filtered :</td></tr>
<tr><td rowspan="2">The precipitate may contain—
HgS—*black*
PbS—*black*
Bi_2S_3—*black*
CuS—*black*
CdS—*yellow*
} Insoluble in KHO and in Am_2S.
SnS—*brown*
SnS_2—*yellow*
Sb_2S_3—*orange*
As_2S_3—*yellow*
[Au_2S_3—*black*]
[PtS_2—*black*]
} Soluble in KHO and in Am_2S.
Examine the precipitate *at once* by Table II. (435).
Note 1.—Any precipitate formed on boiling the filtrate will have to be added to the above.
Note 2.—Unless the liquid has been sufficiently diluted some of the above metals may be afterwards precipitated by Am_2S in Group III., and cause much confusion.</td><td>The solution or filtrate is di- cipitated : as soon as H_2S dish until it ceases to smell and the residue gently ignited heated, then water is added;
Add a few drops of the acid tate shows the *presence of a*
Add to the rest of the HCl solu- any precipitate which may (429): if a phosphate is phates are absent proceed as
[*Note.*—Refer to</td></tr>
<tr><td>The precipitate may contain—
Al_2Ho_6—*almost colourless.*
Cr_2Ho_6—*light green.*
Fe_2Ho_6—*reddish brown.*
Examine the precipitate by Table III. A (436).</td></tr>
</table>

NOTES ON THE GENERAL TABLE.

420. If the liquid under examination is alkaline or neutral in reaction, before commencing the examination by the General Table, HNO_3 must be added in slight excess ; if no precipitate forms, proceed at once to examine the liquid by the General Table ; if a precipitate is produced, more HNO_3 is to be added, and the liquid heated ; should

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* By the "original solution" is meant the solution of the substance if a solid, or the substance itself if a liquid, to which no reagent has been added.

METALS INTO GROUPS BY GROUP-REAGENTS.

requires special treatment (420); if its reaction is *acid*, it may be at once

posed that all metals are present: if a metal or group of metals is absent, no
hence no filtration is necessary, the solution being then treated as a filtrate.

continue to add HCl as long as it causes any further precipitate (421), warm and filter:

tate on addition of several drops of HCl, is boiled down if necessary (423), and is
it is then saturated with H_2S by passing the gas through the solution* (534) for

luted and H_2S is passed again into it, to make sure that Group II. is completely pre-
produces no further precipitate the clear solution or filtrate is boiled in a porcelain
of H_2S (427), a little strong HNO_3 is then added, and the liquid is evaporated to dryness
in the dish. When the dish is cool, a little strong HCl is poured upon the residue and
any undissolved residue is filtered off (428).

solution to some $AmHMoO_4$ solution in a test-tube, and warm gently; a yellow precipi-
phosphate; if no precipitate is formed with $AmHMoO_4$, *phosphates are absent.*

tion AmCl, heat it to boiling, then add 'AmHO in excess, boil again and filter off
form as quickly as possible, keeping the funnel closely covered with a glass plate
present and a precipitate is produced by AmHO refer to Table III.D. (466), if phos-
is directed below:—

(463, *et seq.*), for full directions for precipitating Groups III.A and III.B.]

Add to the filtrate or solution, which may be coloured (480), Am_2S in excess, boil and filter:—

The precipitate may contain— ZnS—*white*. MnS—*light-pink*. CoS—*black*. NiS—*black*. Examine this precipitate *at once* by Table III.B (437).	Add to the filtrate or solution, which must be distinctly yellow or brown (431), Am_2CO_3, warm gently and filter:—	
	The precipitate (432) may contain— $BaCO_3$—*white*. $SrCO_3$—*white*. $CaCO_3$—*white*. Examine the precipitate by Table IV. (438).	The filtrate may contain Mg, K, Na. Examine by Table V. (439).

a precipitate still remain, it is filtered off and the filtrate examined by the General Table, commencing with the addition of HCl.

The precipitate caused and not dissolved by HNO_3 may be finely divided sulphur, separated as a *white* powder from a sulphide, or *yellow* from a thiosulphate†; this precipitate is recognised by its colour, and by not being separable by standing or filtration, it may be disregarded; there may also be precipitated H_4SiO_4 (gelatinous, almost colourless), SnO_2 or metastannic acid (see 420 *a*), Sb_2O_5, AgCl (all three white), SnS_2, As_2S_3 (both yellow), Sb_2S_3 (orange). Hence a full examination of this precipitate for all that it may contain will involve boiling the precipitate with aqua regia, diluting and filtering; the insoluble residue is examined as a substance insoluble in water and acids (475) and the aqua regia solution is added to the filtrate from the precipitate caused by HNO_3, any precipitate caused on thus mixing the solutions being examined by Table I. (433).

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* Instead of diluting and passing the gas, H_2S-solution may be added.

† Formerly called a "hyposulphite."

420 *a*. Metastannic acid is precipitated from a solution of an alkaline metastannate by addition of an acid: it forms a white precipitate which becomes *golden yellow* when moistened with $SnCl_2$ solution; it is further recognised by dissolving in the washing water whilst being washed, and being reprecipitated when the washings run into the acid filtrate.

421. Even if HCl has been used in dissolving the substance, it is advisable to add a few drops of the acid to make sure that no member of Group I. remains.

422. It must be remembered that strong HCl also precipitates a strong solution of a barium salt; this precipitate however disappears, as $PbCl_2$ does, on adding water and heating, thus differing from AgCl and Hg_2Cl_2.

423. If HNO_3 or aqua regia has been used in dissolving the substance, or if the solution smells of Cl or SO_2, it should be boiled down considerably until after adding HCl and boiling, neither of the above gases is smelt; it is then diluted, and H_2S is passed at once whether the dilution has caused a precipitate or not.

If As has been detected in the preliminary examination, H_2SO_3 should be added until the liquid smells of SO_2 after having been boiled for a short time: the liquid is then heated for some time short of boiling until it no longer smells of SO_2. This treatment reduces arsen*ic* and stann*ic* compounds to the arseni*ous* and stann*ous* condition.

In the case of As this reduction is to be recommended, since arsen*ic* compounds are not easily precipitated by H_2S; it is also very desirable to reduce stannic compounds, since SnS_2, unlike SnS, readily runs through the filter, and being yellow in colour may be mistaken for S. It will of course be necessary to ascertain by special tests (187–190, and 201–203), made on the *original solution*, in which state As and Sn were originally present. If any precipitate is produced by boiling with H_2SO_3 other than white sulphur, refer to (424).

424. While boiling with H_2SO_3 some H_2SO_4 is usually formed, which may partially or completely precipitate Pb, Ba, Sr as white sulphates; H_2SO_3 will also possibly precipitate Au, which causes the cool liquid to appear blue by transmitted and red by reflected light, and on boiling separates as a black powder. Examine any precipitate formed by H_2SO_3 by Table G (434).

425. On addition of water, Bi, Sb, Sn may give white precipitates of their oxychlorides; these precipitates may be disregarded, since H_2S readily converts them into sulphides.

426. H_2S often causes a fine *white* precipitate of S, owing to the presence of certain oxidising substances, such as Cl, HNO_3, $HClO_3$, H_2CrO_4, H_2MnO_4, $HMnO_4$, H_2SO_3, Fe_2Cl_6. This precipitate is known by its perfect whiteness, and by not being separable by standing or filtration; it may be neglected. Great care must however be taken not to mistake SnS_2 for S; it resembles S in running through filter paper, but is distinctly *yellow* in colour; it may often be coagulated by being shaken or heated.

426 *a*. Certain changes may be observed whilst H_2S is being passed, they should be carefully noted. Thus Pb and Hg during precipitation by H_2S often show characteristic colours (149, 144); a solution coloured reddish yellow by H_2CrO_4 becomes green; and a solution coloured green by H_2MnO_4 or purple by $HMnO_4$ becomes colourless.

427. If on boiling the filtrate or solution after passing H_2S a yellow precipitate forms, this shows the presence of an arsen*ic* or a stann*ic* compound; in this case pass H_2S into the boiling liquid as long as it causes any further precipitate, or proceed as is directed in the latter part of (423); filter off and add this precipitate to any precipitate already obtained by H_2S to be examined by Table II.

428. SiO_2 and other substances may be present in this residue.

SiO_2 is distinguished by readily dissolving in hot KHO solution.

Al_2O_3, Fe_2O_3, Cr_2O_3 may remain undissolved by HCl, if they have been rendered difficultly soluble by the ignition to which the residue has been subjected; they may be dissolved by long continued heating with strong HCl; the solution is added to the other solution which is to be examined for Group III. A.

$BaSO_4$, $SrSO_4$ may also remain undissolved; they are detected by heating some of the residue strongly for a short time in the inner blowpipe flame upon a loop of platinum wire, then moistening the loop with a drop of strong HCl and holding it in the outer part of the Bunsen flame; crimson red flashes, appearing red through the indigo prism prove the presence of Sr, a yellowish green flame coloration shows Ba. These flame colorations should also be examined by the spectroscope (42).

The following is a less rapid method of detecting Ba and Sr. Dry the insoluble residue, fuse it on platinum foil with three or four times as much fusion mixture; allow it to cool, then boil with water until the mass is disintegrated, filter, wash the residue well upon the filter, and examine it by Table IV. (438) for Ba and Sr only.

429. Mn and Zn are liable to be precipitated with $Fe_2H_6O_6$, $Al_2H_6O_6$, $Cr_2H_6O_6$. The precipitation of Mn is partly prevented by keeping the liquid from exposure to the air after adding AmHO: but the further precaution should be taken of dissolving the precipitate produced by AmHO in HCl and reprecipitating it by AmHO, then filtering and adding the filtrate to the original filtrate from the Group III. A. precipitate; this treatment of the precipitate should be repeated several times, if small quantities of Zn or Mn have to be looked for in presence of Al or Fe; or better still Groups III. A. and III. B. may be precipitated together (459) and the precipitate examined by Tables III. C. (464, 465). See rules given in (463).

430. The filtrate or solution, after the addition of AmCl and AmHO, may be coloured *blue* by Ni, *brown* by Co, or *reddish violet* by $Cr_2H_6O_6$ dissolved in the excess of AmHO.

The $Cr_2H_6O_6$ must be removed before proceeding to examine for the remaining Groups; it is readily separated as a pale green flocculent precipitate by boiling the liquid for a short time in a porcelain dish, adding a little more AmHO if the liquid does not smell of NH_3 after being boiled for a short time; this precipitate is filtered off, and the filtrate is further examined for Groups III. B, IV., and V. by the General Table.

The colorations due to Ni and Co differ from that caused by $Cr_2H_6O_6$ in not being removed by boiling the liquid.

431. A *brown* or *dark coloured* filtrate shows that Ni is present*, some of the NiS precipitated by Am_2S having been dissolved in the excess of Am_2S and giving the filtrate its dark colour. If this coloration is noticed the filtrate should be poured into a porcelain dish and boiled, occasionally adding water if necessary, until on removing the lamp for a short time the black NiS settles, leaving a colourless liquid. The liquid is then run through a small filter and the black precipitate is tested for Ni by fusing a portion of the filter paper stained by the NiS in a colourless borax bead: if a bead is obtained, which when heated in the outer flame is *violet* or *brown* whilst hot and *yellow* when cold, and *which becomes grey or opaque in the inner flame*, Ni is certainly present and need not be further tested for in Table III. B.

The colourless filtrate is examined by the General Table for Groups IV. and V.

432. Part of the precipitate produced by Am_2CO_3 often adheres firmly to the inside of the vessel in which it has been produced, in this case rinse the tube out several times with distilled water and then dissolve the precipitate by pouring in some hot $H\bar{A}$, causing it to run over the inside of the tube, and add this solution to the $H\bar{A}$ solution of the Am_2CO_3 precipitate made in Table IV.

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* If Groups III. A. and III. B. have been precipitated together by AmCl, AmHO, and Am_2S added in succession (459), the brown coloration of the filtrate may also be due to $Cr_2H_6O_6$: on boiling the filtrate coloured by $Cr_2H_6O_6$ in a porcelain dish for several minutes, *pale green* $Cr_2H_6O_6$ is precipitated, whcih cannot be mistaken for black NiS.

433. TABLE I.—SILVER GROUP.

The precipitate produced by HCl may consist of $PbCl_2$, AgCl, and Hg'_2Cl_2: the precipitate on the filter is washed five or six times with boiling water, the first portions of the washings being kept apart :—

The first portions of the washings may contain $PbCl_2$ in solution, which if present in any quantity will be deposited in brilliant crystals on cooling. Test for, or confirm the presence of Pb, by adding to the washings K_2CrO_4: a yellow precipitate, readily soluble in KHO, shows :— *Presence of Pb.*	*Residue:* the residue is rinsed into a test-tube (33 *a, f*) and boiled with AmHO, then filtered :—	
	Filtrate: add HNO_3 until the liquid is acid: a white precipitate uniting into curdy particles when shaken or boiled shows :— *Presence of Ag.*	*Residue* on the filter is black; dry at a gentle heat; scrape the precipitate off the filter, mix with some dry Na_2CO_3, and heat in a small ignition tube (147). Globules of Hg are seen on the sides of the tube, but often only when the inside of the tube is rubbed with a rod or examined with a lens:— *Presence of Hg'* (Mercurosum).

434. TABLE G.—EXAMINATION OF THE PRECIPITATE FORMED ON BOILING THE HCl SOLUTION WITH H_2SO_3. (See par. 424.)

The precipitate produced by H_2SO_3 may contain Au, $PbSO_4$, $BaSO_4$, and $SrSO_4$; the last three substances are perfectly white, and Au, if present, is therefore usually seen by colouring the precipitate brown or black; the Au is also usually visible during precipitation by H_2SO_3, or after the white sulphates have subsided, since it colours the liquid blue by transmitted and reddish by reflected light.

Ba and Sr can often be at once detected, if present, by taking a little of the moist precipitate on a loop of platinum wire, heating in the inner blowpipe flame for some time, moistening with HCl, and examining the flame coloration; but a more trustworthy method of proceeding is the following:—

Rinse the precipitate into a small porcelain dish, using as little water as possible; dissolve in the liquid a small crystal of $H_2\bar{T}$ or add a few drops of strong $H\bar{A}$; then add AmHO until it is just in excess, and boil for a short time, let stand and decant through a filter :—

Solution may contain $PbSO_4$; add $H\bar{A}$ in excess, then K_2CrO_4; a yellow precipitate soluble in KHO shows :— *Presence of Pb.*	*Residue:* pour upon the residue in the dish a little HCl and a few drops of HNO_3, heat to boiling, let stand, decant into a porcelain dish :—	
	Solution: boil until the smell of Cl is removed; add several drops of fresh $FeSO_4$ solution; a blue coloration and reddish precipitate show:— *Presence of Au.*	*Residue:* dry by gently heating the dish, fuse with fusion mixture on platinum foil, and examine the mass for Ba and Sr as directed in the last part of (428).

These lines run across both pages.

435. TABLE II.—COPPER AND

The precipitate produced by H_2S in the HCl solution, after having with KHO or a small quantity of Am_2S*, and filtered :—

GROUP II. A.—COPPER GROUP.

<table>
<tr><td colspan="5">435. <i>a. The residue</i> may contain HgS, PbS, Bi_2S_3, CuS, CdS. After having washed the precipitate and allowed it to drain, remove it from the filter into a porcelain dish (33 <i>d</i>), pour upon it strong HNO_3 sufficient to cover it completely, and heat gently as long as any red fumes come off, adding more strong HNO_3 if necessary to prevent evaporation to dryness. Boil off nearly all the acid, add a little dilute H_2SO_4, let stand for some time, stirring occasionally, and filter :—</td></tr>
<tr><td colspan="2" rowspan="2"><i>Residue</i> may contain HgS (<i>black</i>), $PbSO_4$ (<i>white</i>); but if <i>white</i>, the absence of Hg is not proved.
Remove the residue into a porcelain dish, using as little water as possible; add a little $H_2\bar{T}$, then AmHO in excess, boil and filter :—</td><td colspan="3"><i>Filtrate</i> may contain Bi, Cu, Cd.
Add AmHO in excess, boil and filter :—</td></tr>
<tr><td rowspan="2"><i>Precipitate</i> (not easily seen in dark blue solutions): wash with hot water, dissolve off the filter by pouring upon it <i>a few drops</i> of boiling dilute HCl, pour this solution into a large quantity of cold distilled water, a milkiness appearing at once or after stirring and letting stand for some time, shows :—
<i>Presence of Bi</i>.
<i>Note.</i>—A precipitate produced by AmHO must always be tested as above for Bi, since Pb and Hg might possibly appear here as white precipitates and be mistaken for Bi.</td><td colspan="2"><i>Filtrate, if blue, contains Cu</i>: (note 2) add HCl until the liquid is acid, then pass H_2S to saturation, filter and wash quickly, keeping the filter covered with a glass plate; rinse the precipitate into a test-tube, pour upon it some dilute H_2SO_4, boil and filter through a covered filter (note 3):—</td></tr>
<tr><td><i>Residue</i>: dry on the filter at a gentle heat, best in the steam oven. Scrape the dried residue off the filter (note 1), and heat it, mixed with dry Na_2CO_3, in a small ignition-tube; globules of Hg form on the sides of the tube, which become visible when rubbed or when looked for by a lens :—
<i>Presence of Hg''</i> (Mercuricum).</td><td><i>Filtrate</i>:
Add $H\bar{A}$ in excess, then K_2CrO_4, a yellow precipitate :—
<i>Presence of Pb</i>.</td><td><i>Residue</i>: dissolve by pouring a little boiling HNO_3 upon the filter, add to the solution AmHO in excess, then $H\bar{A}$ in excess and a few drops of K_4FeCy_6: a chocolate-red precipitate shows :—
<i>Presence of Cu</i>.</td><td><i>Filtrate</i>: add much water and pass H_2S for some time, or add much H_2S-water, a yellow precipitate shows :—
<i>Presence of Cd</i>.
<i>Note.</i>—If this precipitate is dark-coloured, CuS is present (see note 3): filter it off, boil it once more with H_2SO_4; filter and test the filtrate by H_2S as above, for Cd.</td></tr>
</table>

*** If traces of Cu have to be tested for, Na_2S should be substituted for Am_2S, since CuS is**

Note 1.—If the residue is too small to be removed, the portion of the paper containing it may be cut up and mixed with Na_2CO_3. For the precautions requisite in performing this test see (147).

Note 2.—Another method of detecting Cd *in the presence of Cu*, consists in adding KCy solution to the blue ammoniacal liquid, and passing H_2S ; Cd if present falls as yellow sulphide ; Cu may then be tested for in the filtrate which has been boiled down considerably with excess of HNO_3, by adding AmHO in excess, then $H\bar{A}$ in excess, and K_4FeCy_6.

Note 3.—Unless this precipitate is filtered without delay in a covered funnel, washed quickly, and examined at once, CuS is liable to be oxidised and to

ARSENIC GROUPS.

been washed with hot water, is removed from the filter and boiled

GROUP II. B.—ARSENIC GROUP.

The filtrate **may contain As_2S_3, Sb_2S_3, SnS_2, SnS, [Au_2S_3, PtS_2]. Add to it HCl until it is acid, and pass H_2S unless the liquid smells strongly; the above sulphides if present will be reprecipitated; a mere milkiness due to precipitation of S may be disregarded.**

The precipitate is filtered off and examined as directed in 435 *b* (see note 4, below):—

435 *b*. Remove the precipitate from the filter into a porcelain dish, using as little water as possible; add a little strong HCl and heat, occasionally dropping in a crystal of $KClO_3$, until the precipitate dissolves entirely or leaves only a slight residue of sulphur. Heat this solution just short of boiling (note 5) until it ceases to smell of Cl (note 6), then cool it and pour it, through a filter if necessary, into a small flask (181) containing pieces of pure Zn: if the gas does not come off readily, add a little strong HCl, and cause the gas after passing through some $Pb\bar{A}_2$ solution (see caution 181) to bubble through some $AgNO_3$ solution contained in a broad test-tube. As soon as the evolution of gas slackens, drop in more Zn, unless some remains undissolved in the flask, and as soon as the gas ceases to come off, some Zn still remaining undissolved, remove the test-tube containing the $AgNO_3$ solution; if any black precipitate has formed in this solution it must be examined as below; if the liquid has remained clear and colourless it may be rejected, since As and Sb are absent.

Residue **in the flask may consist of Sn, (Pt,Au), and excess of Zn; remove any deposit of Sn by shaking the pieces of Zn well with the liquid and quickly pouring off into a dish, decant the liquid and wash the Sn by decantation in the dish: boil the residue with a little strong HCl in a test-tube, then quickly dilute, and decant; [the residue may contain Au, Pt, see (474)]: add $HgCl_2$ to the solution; a white precipitate, becoming black with AmHO, shows:—** ***Presence of Sn.*** **If H_2S has not proved in the General Table (427), whether Sn'' or Sn'''' is present, refer to (201–203).**	*To the liquid in the test-tube* add several drops of $AgNO_3$ solution, filter:—	
	Precipitate: wash well with boiling water until HCl gives no milkiness with the last few drops of the washing water, then pour upon the filter a little boiling dilute $H_2\bar{T}$ solution, boil it and pour it again through the filter; repeat this several times, then add to the solution HCl and pass H_2S; an orange red precipitate shows:— *Presence of Sb.* *Note.*—This precipitate is often only seen on settling from the yellow liquid.	*Filtrate:* dilute several drops of AmHO largely in a test-tube and add this liquid drop by drop to the filtrate: a light yellow precipitate, which settles in flocks on shaking the liquid shows:— *Presence of As.* If H_2S has not proved in the General Table (427), whether As''' or As''''' is present, refer to (187–190).

somewhat soluble in Am_2S; but $Sn''S$ and Au_2S_3 are much more readily soluble in Am_2S.

pass into the filtrate as $CuSO_4$; it then discolours the CdS precipitate, or may be entirely overlooked if present in small quantity. It is well to pour a few drops of H_2S-water into the funnel with each fresh addition of washing water.

***Note* 4.**—The detection of As, Sb, Sn by the method given in (435 *b*) is very trustworthy and delicate (208); it is excelled in these respects by the method in (210). For beginners, however, the process (209) is recommended by its greater simplicity.

***Note* 5.**—The solution must not be heated to boiling, else $SbCl_5$ or $SnCl_4$ if present might be volatilised.

Note 6.—In this solution As and Sn will be present as stann*icum* and arsen*icum*, and in this state they are not well suited for detection: the following modification makes their detection much more rapid and certain, especially when they are present only in small quantity. After warming until the liquid ceases to smell of Cl, add H_2SO_3 drop by drop, until the liquid after being warmed for a short time smells of SO_2; then heat it short of boiling until the smell of SO_2 is removed, and pour, through a filter if necessary, into the hydrogen apparatus. Great care must be taken to remove the SO_2 entirely, else Sb and As may remain as sulphides in the hydrogen apparatus.

436. TABLE III.A.—IRON GROUP.

The precipitate produced by boiling, after addition of AmCl and of AmHO in excess, may contain Al_2Ho_6, Cr_2Ho_6, Fe_2Ho_6: the colour of the precipitate will usually show whether it contains any quantity of Fe_2Ho_6 (reddish brown), or Cr_2Ho_6 (pale green), since Al_2Ho_6 is colourless.

Dissolve the precipitate in *a little* boiling dilute HCl (33 *d*, *e*, *f*), add to the solution pure KHO or NaHO until the precipitate remains after stirring, then add KHO or NaHO in considerable excess, boil for some time and filter:—

<table>
<tr>
<td rowspan="2">1. <i>Filtrate</i> may contain Al_2Ho_6 dissolved in excess of NaHO: if it is green, Cr_2Ho_6 is also in solution and must be precipitated by further boiling in a porcelain dish and then removed by filtration.
Add AmCl solution in excess to the colourless filtrate: a colourless gelatinous precipitate shows:—
<i>Presence of Al.</i>
<i>Note.</i>—Sodium and potassium hydrates are very liable to contain Al_2Ho_6: the analyst must ascertain that the Al does not come from this source.</td>
<td colspan="2">2. <i>Precipitate</i> may contain Cr_2Ho_6 and Fe_2Ho_6.
Dry upon the filter, carefully avoiding charring the paper; when sufficiently dried the substance will have shrunk considerably into small, hard, dark-coloured pieces; detach these from the filter and fuse them with a little solid Na_2CO_3 and KNO_3 upon a piece of platinum foil, either supported upon a pipeclay triangle or held in the Bunsen flame by crucible tongs. Continue the fusion by heating the under surface of the foil in the blowpipe flame as long as any frothing occurs, then allow to cool; a yellow mass indicates the presence of Cr (note 1). Boil the platinum foil in a small porcelain dish with water until the substance is entirely dissolved or only a dark brown powder (Fe_2O_3) remains undissolved; take out the foil, allow the powder to settle, and decant the liquid through a filter:—</td>
</tr>
<tr>
<td>3. <i>Filtrate</i> will be yellow if Cr is present (note 1<i>a</i>).
Add $H\overline{A}$ in excess, shown by the colour becoming reddish, boil for several minutes, then add $Pb\overline{A}_2$ solution; a yellow precipitate shows:—
<i>Presence of Cr</i> (note 2).</td>
<td>4. <i>Residue</i> in porcelain dish; dissolve by pouring in a little HCl and boiling, add some water, then a few drops of KCyS solution; a blood-red coloration shows:—
<i>Presence of Fe</i> (note 3).</td>
</tr>
</table>

Note 1.—If Mn was present in the original substance, it is often precipitated more or less completely with the Fe, and will be detected by imparting to the fused mass a bluish green colour; this should be noted, as the Mn may possibly be entirely precipitated in this group, and will then not be detected in the next group.

Note 1 *a.*—The green colour, due to the presence of Mn, conceals the pale yellow colour due to Cr; but on dissolving the cool mass in water and boiling with $H\bar{A}$, the manganese coloration is destroyed; and, on filtering from any dark coloured precipitate, the yellow colour due to Cr becomes evident.

Note. 2.—Cr may have been present as a chromic salt, Cr replacing hydrogen in an acid; or as a chromate, Cr being united with oxygen to form an acid-radicle. Ascertain in which state it was present by boiling some of the original substance with Na_2CO_3 solution and filtering; a yellow filtrate, which on being acidified with $H\bar{A}$ gives a yellow precipitate with $Pb\bar{A}_2$, shows that Cr was *present as a chromate;* Cr previously combined with an acid as a chromic salt, remains undissolved as green $Cr_2H_6O_6$, and does not colour the filtrate: it is found by 2 in the preceding Table.

Note 3.—Fe is always detected here as a ferric salt, since, even if originally present as a ferrous compound, it will have been oxidised by boiling the solution with HNO_3 before precipitating Group III.A. in the General Table. To ascertain in which condition Fe was present, boil some of the original substance with HCl, filter if necessary, and divide the solution into two parts: to one portion add $K_6Fe_2Cy_{12}$, a dark blue precipitate shows the *presence of* Fe'' (*Ferrosum*); to another portion add KCyS, a blood red colour shows *presence of* Fe''' (*Ferricum*).

437. TABLE III.B.—ZINC GROUP.

The precipitate produced by Am_2S in the solution, or in the filtrate from Group III.A., may contain ZnS, MnS, NiS, CoS. The colour of the precipitate will usually show whether it contains any quantity of NiS or CoS which are black, or consists only of MnS (pink) or ZnS (white).

The presence of Ni will have been indicated by the filtrate obtained after boiling with AmHO in the General Table being blue in colour, and by a dark brown filtrate having been obtained after boiling with Am_2S. If the black precipitate, obtained by boiling the dark brown filtrate from Am_2S (431), has also been proved by the borax bead to be NiS, the further tests for Ni in the following Table may be omitted.

Remove the precipitate from the filter with cold dilute HCl in the way described in par. 33 *c;* take out the paper and allow the liquid to stand, occasionally stirring it well. The precipitate will either dissolve, leaving only white sulphur, in which case the milky liquid may be at once examined by 2 (p. 247), or a black residue will be left which must be filtered off and examined by 1 (below), the filtrate being examined by 2 (p. 247) :—

<table>
<tr><td colspan="6">1. <i>The Residue</i> is black, and may contain NiS, CoS.
<i>Examination by the borax bead.</i>
Fuse a small quantity in a clear borax bead in the extreme tip of the outer blowpipe flame, note its colour; then fuse the bead for some time in the inner blowpipe flame, and again observe its colour. Any one of the following observations may be made :—</td></tr>
<tr><td colspan="2"><i>Blue bead</i> in both flames shows :—
<i>Presence of Co.</i>
Co need not be further tested for, but if any doubt exists as to the presence of Ni the rest of the residue must be examined as below.</td><td colspan="2"><i>Brown or yellow bead,</i> when cold after fusing it in the outer flame, which becomes <i>grey or opaque</i> in the inner flame, shows <i>Presence of Ni, Absence of Co.</i>
Unless mere traces of Co have to be tested for, no further examination need be made.</td><td colspan="2"><i>A bead which is neither brown nor blue,</i> but of some intermediate hue on cooling from the outer flame, indicates the probable presence of both Co and Ni.
Examine the remainder of the black residue as directed below for Ni and Co, or for Co alone if Ni has been already detected by (431).</td></tr>
<tr><td colspan="6"><i>Further examination of the residue.</i>—Rinse the residue from the filter into a porcelain dish, using as little water as possible: pour in a little strong HCl and boil for some time, adding occasionally a small crystal of $KClO_3$, until the black residue is entirely dissolved or only a small quantity of dark sulphur remains; then boil down nearly to dryness, a blue liquid shows <i>presence of Co;</i> dilute with a little water, filter if necessary into a boiling-tube and pour in KCy solution slowly until the precipitate formed at first is just redissolved, boil briskly for several minutes, and add much strong Na_2Cl_2O, or make decidedly alkaline with NaHO and add much Br-water; heat nearly to boiling, and allow the liquid to stand at least ten minutes, filter* :—</td></tr>
<tr><td colspan="3"><i>Precipitate (black)</i>: wash, and confirm the presence of Ni by fusing some of the precipitate, or the paper stained with the precipitate, in a borax bead in the outer and inner blowpipe flames: a bead <i>yellow</i> when cold, becoming <i>black</i> in the inner flame shows :—
<i>Presence of Ni.</i></td><td colspan="3"><i>Filtrate</i> must be warmed with more Na_2Cl_2O or Br-water, and filtered from any additional precipitate which may form, then evaporated to dryness and strong HNO_3 poured upon the residue as long as any frothing is caused; this is then evaporated just to dryness, the residue dissolved in water, excess of KHO added to it, and any precipitate filtered off and fused into a borax bead, taking the paper stained by the precipitate if the precipitate is not easily removed: a blue bead shows :—
<i>Presence of Co.</i></td></tr>
</table>

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* If this precipitate adheres to the sides of the boiling tube, it is dissolved, after the liquid has been emptied out, by pouring in a little boiling HCl; from this solution the Ni is precipitated by addition of KHO, and the precipitate filtered off and tested by the borax bead for Ni.

2. *The Solution or filtrate* may contain Zn, Mn. Boil in a porcelain dish until it ceases to smell of H_2S, then drop in a small crystal of $KClO_3$, and boil for several minutes. After cooling the liquid add pure NaHO until after stirring the liquid it turns red litmus-paper blue, then add more NaHO, stir well and filter:	
Precipitate is white at first, but rapidly darkens in the air. Fuse a portion of it with solid Na_2CO_3 and KNO_3 on platinum foil; a bluish green mass is obtained on cooling, showing: *Presence of Mn.* *Note.*—Mn may have been originally present either replacing H in an acid, or combined with O as an acid-radicle. Its presence in an acid-radicle is indicated by the colour of the original solution, since in the form of manganate it is green, and as permanganate it is purple; these colours disappear on boiling the HCl solution for some time, or on passing H_2S.	*Filtrate;* pass H_2S, a white precipitate forms, often somewhat discoloured, showing: *Presence of Zn.* *Note.*—From a dilute solution of $ZnHo_2$ in NaHO the $ZnHo_2$ is precipitated on boiling; hence after adding NaHO, as directed above, the liquid must not be boiled, else Zn might be precipitated with the $MnHo_2$ and escape detection. But by diluting and boiling the above filtrate it is usually possible to cause any $ZnHo_2$ it holds in solution to precipitate, especially if the alkalinity of the liquid be somewhat reduced by cautious addition of HCl in quantity insufficient to make the liquid acid. If this precipitate be separated by pouring the liquid through a double filter and then be dissolved off the filter and the sides of the boiling tube by a little boiling $H\bar{A}$, ZnS may be precipitated *perfectly white* from this solution by H_2S.

438. TABLE IV.—BARIUM GROUP.

The precipitate produced by Am_2CO_3 may contain $BaCO_3$, $SrCO_3$, $CaCO_3$: it is to be well washed with boiling water. Before dissolving the precipitate off the filter much may usually be learnt by an examination of the flame coloration yielded by the HCl solution (see 1 below). The results thus obtained are afterwards confirmed in the wet way (see 2 below).

1. *Examination by flame coloration.*—Take a small quantity of the precipitate off the filter upon the end of a glass rod, and dissolve it by moving the end of the rod about in several drops of HCl on a watch glass; dip into this solution a loop of platinum wire which imparts no colour to the flame, then hold the loop for some time in the Bunsen flame, repeating the process if the coloration is not satisfactorily observed at first. Ca will impart to the flame a *yellowish red* colour, Sr a *crimson red,* Ba a *yellowish green.* The red colorations yielded by Ca and Sr are distinguished by viewing the flame through the indigo-prism; the Ca

e lines across pages.

coloration then appears *dingy green*, whilst the Sr coloration still
ing after the others have disappeared. When all three metals
one another; but if at any time a red colour is visible through
visible without using the prism, the presence of Ba is proved. The
2. *Examination in the wet way.*—The rest of the precipitate
again boiling the liquid and pouring it once more through the
the solution is acid, and if it is not, add $H\bar{A}$ whilst stirring until
solution into a test-tube, and put by the larger portion (*B*). To
part of the tube in cold water or holding it in a stream from the
cipitate forms refer to Column I. (below): if no immediate pre-
forms refer to Column II., if no precipitate forms even after

<table>
<tr><td colspan="3">I. <i>An immediate precipitate is produced by $CaSO_4$ in the cold, showing Presence of Ba.</i>
Portion (<i>B</i>) must then be tested for Sr and Ca which may also be present: examine it as directed below :—
Add K_2CrO_4 solution until the colour of the liquid is reddish yellow; heat, and filter through a double filter-paper, pouring the filtrate through the same filter repeatedly if necessary until the liquid is quite clear; then add to the liquid, which must be orange red in colour, AmHO until the colour changes to pale yellow, then add Am_2CO_3 solution :—</td></tr>
<tr><td rowspan="2"><i>No precipitate forms,</i> showing :—
<i>Absence of Sr and Ca.</i></td><td colspan="2"><i>A precipitate forms,</i> showing Sr, Ca, or both of them, to be present. Add to the liquid Am_2CO_3 in excess, filter, reject the filtrate and dissolve the precipitate off the filter in as little boiling $H\bar{A}$ as possible; pour off a small part (<i>A'</i>) of this solution, reserving the larger portion (<i>B'</i>). To (<i>A'</i>) add $CaSO_4$ solution and boil :—</td></tr>
<tr><td><i>No precipitate forms:—</i>
<i>Absence of Sr.</i>
Examine portion (<i>B'</i>) for Ca as directed at (<i>C</i>) in Columu III.</td><td><i>A precipitate forms:—</i>
<i>Presence of Sr.</i>
Examine portion (<i>B'</i>) for Ca as directed at (<i>C</i>), Column II.</td></tr>
</table>

439. TABLE V.—

lines cross pages.

The filtrate remaining after addition to the original solution of all
NH_4. Since, however, NH_4-salts have been added as group-
be detected by boiling a portion of the original substance with
which would be recognised by its smell or its action on moistened
here for NH_4, since it will have been already tested for in the
as directed below.

appears *crimson;* Ba is usually found by its coloration remain-
are present together their colorations are apt to interfere with
the indigo-prism, Sr is certainly present; if a green coloration is
flame coloration should be examined by the spectroscope (42).
is dissolved off the filter by pouring upon it a little boiling $H\bar{A}$,
filter if the precipitate is not entirely dissolved. Test whether
the liquid is acid, then pour off a small portion (*A*) of the acid
the portion (*A*), after it has been cooled by immersing the lower
tap, add several drops of $CaSO_4$ solution; if an immediate pre-
cipitate forms heat the liquid to boiling, and if a precipitate then
several minutes refer to Column III.

II. *A precipitate is not formed at once on the addition of $CaSO_4$, but appears on boiling the liquid, showing:—*	III. *No precipitate is produced by $CaSO_4$, even on boiling, showing:—*
Absence of Ba and Presence of Sr.	*Absence of Ba and Sr, and Presence of Ca.*
C. Portion (*B*) is then examined for Ca as directed below:— Add dilute H_2SO_4 in excess, boil and filter, reject the precipitate; add to the filtrate several drops more H_2SO_4 and boil; if this causes any precipitate boil and filter, and again test the filtrate by addition of H_2SO_4 and boiling: repeat this process if necessary. To the clear filtrate, which gives no further precipitate on addition of H_2SO_4 and boiling, add gradually AmHO until after mixing the liquid by thorough stirring or shaking, it turns red litmus-paper blue, then add $Am_2C_2O_4$ solution and warm gently; a white precipitate, often appearing only after a time, shows:— *Presence of Ca.* (See Note, Column III.)	*C.* Confirm the presence of Ca by making portion (*B*) alkaline with AmHO (34), then add $Am_2C_2O_4$ solution; a white precipitate forms, showing:— *Presence of Ca.* *Note.*—If mere traces of Ca have to be tested for, the filter-paper used in the analysis must first be freed from any traces of Ca it may contain by wetting it with dilute HCl, and then thoroughly washing it with distilled water, as described in the last par. of (29).

POTASSIUM GROUP.
the group-reagents in succession, may still contain Mg, K, Na, and
reagents, it is useless to test for NH_4 in this filtrate: NH_4 must
KHO-solution, and ascertaining whether any NH_3-gas is evolved,
red litmus-paper. It is, however, usually unnecessary to test
preliminary examination (390). Proceed to examine for Group V.

Evaporate the filtrate from Group IV. to dryness in a porcelain dish, scrape out the solid residue and heat it to redness upon a piece of platinum foil in the Bunsen flame as long as any white fumes are seen to be given off on removing the foil for an instant from the flame (note 1). All NH_4-compounds are thus entirely removed. If any residue remains (note 2) it is to be dissolved by boiling the foil in a test-tube with a small quantity of water to which several drops of dilute HCl have been added. Divide this solution into two parts :—

<table>
<tr><th>EXAMINATION FOR Mg.</th><th colspan="2">EXAMINATION FOR K AND Na.</th></tr>
<tr><td rowspan="3">Before testing for Mg in one portion of this solution it is necessary to remove from it any traces of Ba, Sr, or Ca which it may possibly contain,* and which might else be mistaken for Mg.
Add, therefore, several drops of H_2SO_4 to the liquid, boil for a short time and let stand ; then, whether a precipitate has formed or not, add a little AmCl, then AmHO in excess, then several drops of $Am_2C_2O_4$, and warm gently ; if any precipitate has formed proceed to filter at once as quickly as possible. To the liquid, in which the addition of H_2SO_4, AmCl, AmHO in excess and $Am_2C_2O_4$ causes no further precipitate, add Na_2HPO_4, and if no precipitate forms at once warm gently, shake or stir the liquid violently and let it stand for some time : a white crystalline precipitate shows :—
Presence of Mg.</td><td colspan="2">In the other portion proceed to test for K and Na as directed below. The presence of Mg in no way interferes with the detection of K and Na, and hence if Mg has been detected its removal is unnecessary.
Flame coloration.—Dip into the solution a loop of platinum wire which has been proved not to impart any colour to the flame. Hold the loop in the Bunsen flame ; one of the following results will be observed :—</td></tr>
<tr><td>A bright yellow flame coloration, indicating the presence of Na (note 3).
Examine this coloration through the indigo-prism ; if it appears red, presence of K is proved : if no red colour is visible K is probably absent or present only in very minute quantity.</td><td>A pale violet coloration, appearing crimson red through the indigo-prism, shows :—
Presence of K, and absence of Na.</td></tr>
<tr><td colspan="2">It is usual to confirm the results of the examination of the flame coloration by pouring the remainder of the solution upon a watch glass, adding to it several drops of $PtCl_4$ (note 4) and stirring well for some time ; the formation of a yellow precipitate shows :—
Presence of K.
See (47), and Note 5, page 251.</td></tr>
</table>

Note 1.—If the residue is small in amount it may be ignited in the dish ; but this is not to be generally recommended, as the porcelain dish is liable to be cracked by the heat, and it is also difficult to get entirely rid of the NH_4-salts by heating only in porcelain.

Note 2.—It is not safe to place much reliance upon an examination of the foil for the detection of a small quantity of residue, but it may usually be detected by its producing a crackling noise whilst the foil is cooling immediately after its removal from the flame. Should there be any doubt, the foil must be boiled with water and a drop of HCl, and the solution examined for Mg, K, and Na, as directed above.

---

* Traces of Ba, Sr, Ca may remain unprecipitated by Am_2CO_3.

Note 3.—The examination of this flame coloration by means of the spectroscope will naturally suggest itself.

A yellow coloration, more or less intense, will almost always be obtained here, since most substances and reagents contain small quantities of Na; hence the student must note the intensity of the coloration, and judge from it whether the quantity of Na is small or large; he must then enter accordingly, either "*presence of Na,*" or "*presence of trace of Na.*"

Note 4.—If iodine is present, $PtCl_4$ will produce an intense red coloration; hence if iodine is suspected to be present, before adding $PtCl_4$ the above solution should be evaporated to dryness with a little strong HNO_3, the residue dissolved in a few drops of dilute HCl and tested with $PtCl_4$ for K. This is of course unnecessary if, before precipitating Group III. in the General Table, the solution has been evaporated to dryness with HNO_3. In case of uncertainty, add $PtCl_4$ to a drop only of the above solution; and if iodine is found to be present proceed as is directed above.

Note 5.—Mere traces of K and Na may be detected by adding $PtCl_4$, and evaporating the liquid to dryness in a porcelain dish upon a water-bath; absolute alcohol is then poured into the dish and stirred; any yellow residue shows *presence of K*, the solution giving the pure Na coloration if Na is present. On filtering off the yellow residue and washing it with absolute alcohol, it will yield the pure K flame coloration.

TABLE OF SOLUBILITY.

A blank signifies that the solubility is unknown or unimportant.

	K.	*Na.*	*Am.*	*Mg.*	*Ba.*	*Sr.*	*Ca.*	*Fe''.*	*Fe'''.*	*Al.*	*Cr.*	*Zn.*	*Mn.*	*Ni.*	*Co.*
Oxide . . .	w.	w.	w.	a.	w.	w.	w-a.	a.	a.	a.i.	a.i.	a.	a.	a.	a.
Sulphide . .	w.	w.	w.	w-a.	w.	w.	w-a.	$a^{m.}$	a.	—	—	a.	a.	$a^{mn.}$	a^{mn}
Chloride . .	w.	w.	w.	w.	w.	w.	w.	w.	w.	w.	a.i.	w.	w.	w.	w.
Iodide . . .	w.	w.	w.	w.	w.	w.	w.	w.	w.	—	—	w.	w.	—	—
Sulphate . .	w.	w.	w.	w.	i.	i.	w-$a^{m.}$-i.	w.	w.	w.	w.a.	w.	w.	w.	w.
Nitrate . .	w.	w.	w.	w.	w.	w.	w.	w.	w.	w.	w.	w.	w.	w.	w.
Phosphate .	w.	w.	w.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.
Carbonate .	w.	w.	w.	a.	a.	a.	a.	a.	—	—	—	a.	a.	a.	a.
Borate . . .	w.	w.	w.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.
Arsenite . .	w.	w.	w.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.
Arsenate . .	w.	w.	w.	a.	a.	a.	a.	a.	a.	—	—	—	—	a.	a.
Chromate . .	w.	w.	w.	w.	a.	a.	a.	—	w.	a.	a.	w.	i.	a.	—
Fluoride . .	w.	w.	w.	a.	a-i.	i.	a-i.	—	—	—	—	—	—	—	—
Oxalate . .	w.	w.	w.	a.	a.	a.	a.	a.	a.	a.	w-a.	a.	w-a.	a.	a.

	Hg''.	*Pb.*	*Bi.*	*Cu.*	*Cd.*	*Sb.*	*Sn''.*	*Sn''''.*	*As'''.*	*Ag.*	*Hg'.*
Oxide . . .	a.	$a^{n.}$	a.	a.	a.	$a^{m.}$	a.	a.i.	w.$a^{m.}$	$a^{n.}$	$a^{n.}$
Sulphide . .	$a^{mn.}$	$a^{n.}$	$a^{n.}$	$a^{n.}$	a.	$a^{m.}$	$a^{m.}$	$a^{m.}$	$a^{n.}$	$a^{n.}$	$a^{n.}$
Chloride . .	w.	w.i.	w.b.	w.	w.	w.b.	w.b.	w.	w.	i.	a.i.
Iodide . . .	a.	w.$a^{m.}$	—	—	w.	—	w.	w.	—	i.	$a^{n.}$
Sulphate . .	w.b.	a.i.	w.b.	w.	w.	a.	w.	w.b.	—	w.$a^{n.}$	w.b.a^{n}
Nitrate . .	w.b	w.	w.b.	w.	w.	—	—	w.	—	w.	w.b.
Phosphate .	a.	$a^{n.}$	—	a.	a.	—	a.	—	—	$a^{n.}$	$a^{n.}$
Carbonate .	a.	$a^{n.}$	a.	a.	a.	—	—	—	—	$a^{n.}$	$a^{n.}$
Borate . . .	—	$a^{n.}$	a.	a.	w.a.	—	a.	—	—	—	w.
Arsenite . .	a.	$a^{n.}$	a.	a.	—	a.	—	—	—	$a^{n.}$	$a^{n.}$
Arsenate . .	a.	$a^{n.}$	—	a.	—	a.	—	—	—	$a^{n.}$	$a^{n.}$
Chromate . .	w-a.	$a^{n.}$i.	a.	w.	—	a.	—	—	—	$a^{n.}$	$a^{n.}$
Fluoride . .	w.	a.	—	—	—	—	—	—	—	w.	—
Oxalate . .	a.	a.	a.	a.	a.	a.	a.	w.	—	a.	a.

The solubility of a compound is denoted by letters :—

w. Signifies soluble in water.

a. Soluble in acids ; the term standing for HCl, HNO_3, and aqua regia.

$a^{m.}$ Soluble in muriatic, or hydrochloric acid.

$a^{n.}$ Soluble in nitric acid.

$a^{mn.}$ Soluble in a mixture of muriatic and nitric acids, or aqua regia, but not in either acid separately.

i. Insoluble in water and acids.

w.a. Letters thus placed together with a stop between, signify that in different states the substance shows the different solubilities denoted by the letters.

w-a. Letters connected by a hyphen indicate that the substance is only slightly soluble in the first solvent, and may therefore partially fall under the class denoted by the second letter.

w.b. Decomposed more or less by much water with formation of a basic salt which is insoluble in water but soluble in acid.

EXAMINATION FOR ACID-RADICLES.

INTRODUCTORY REMARKS.

440. In conducting the examination for acid-radicles much time and trouble may usually be saved by considering which of these radicles can possibly be present. The preceding examination will usually have limited this number considerably (441), but it may be further reduced by the knowledge already obtained of the metals present, and of the solubility of the substance under analysis.

A reference to the Table of Solubility on the preceding page will show how this knowledge may be applied.

Only the most commonly occurring compounds are contained in this table, the solubility of other substances may be obtained by reference to Storer's "Dictionary of Solubilities."

The table is thus arranged: in a horizontal line at the head are placed the more commonly occurring metals, which yield salts or basic oxides: in the vertical column on the left is a list of that portion of the names of the compounds thus formed, which corresponds to the acid-radicle.

To find the solubility of any compound of one of the metals placed at the top of the Table, glance down the vertical column which is headed by this element, the letter indicating the solubility of the compound will be found in a horizontal line with the acid-radicle portion of the name. Thus to find the solubility of zinc sulphate, it is only necessary to glance down the vertical column with *Zn* at its head: on a horizontal line with *sulphate* stands the letter *w*, showing that zinc sulphate is soluble in water.

The way in which this Table is used after ascertaining the solubility of a substance under analysis, and detecting the metals present in it, may be explained by an example. In a substance *which was entirely soluble in water*, the metals found were K, Ba, Ag. A glance down the columns headed by these three metals shows at once which acid-radicles may be present: all acid radicles might be present combined with K since all its salts are soluble in water, but the presence of Ba in a substance soluble in water shows that SO_4, PO_4, CO_3, BO_3, AsO_4, CrO_4, and F cannot be present, since these acid-radicles form compounds with Ba which are insoluble in water: Ag excludes in addition S, Cl, Br, and I; hence amongst the more commonly occurring acid-radicles only NO_3 need be tested for.

This example shows how much the examination for acid-radicles may often be simplified, when the metals present in a substance and its solubility are known.

441. Several acid-radicles will probably have been satisfactorily tested for in the preliminary examinations: in case their reactions have been interfered with by the presence of other substances, the analyst should refer to the reactions given in the fourth section and select one which will be decisive.

The examination for metals also frequently yields proof of the presence of certain acid-radicles. Thus on passing H_2S into the HCl solution:—

A reddish yellow solution, becoming green and depositing white sulphur, shows	*Presence of* $(CrO_4)''$
A green solution, becoming colourless, shows	*Presence of* $(MnO_4)'''$
A purple solution „ „ „	*Presence of* $(MnO_4)'$
A yellow precipitate of As_2S_3, appearing only when the liquid is boiled, shows	*Presence of* $(AsO_4)''$
An insoluble residue of SiO_2, after evaporating to dryness the filtrate from the H_2S-group, shows	*Presence of* $(SiO_3)''$

And a yellow precipitate obtained with $AmHMoO_4$ before precipitating Group III., shows *Presence of* $(PO_4)'''$

The presence of any of these acid-radicles thus detected will require no further confirmation.

Of the acid-radicles which remain to be tested for, some can be detected by the plan drawn out in (442–445); others are most easily found by special tests made on the original substance (446–454).

GENERAL EXAMINATION FOR ACID-RADICLES.

442. Before employing the liquid tests it is advisable to separate from the substance any metals other than K, Na, and NH_4 which it may contain, since some of these are liable to be precipitated by the reagents added for the detection of the acid-radicles. If alkali-metals alone are present this separation is unnecessary, since they are not precipitated by any of the reagents.

This separation of the metals may usually be effected by boiling a portion of the finely-powdered substance with Na_2CO_3 solution, which must be further added to the clear solution as long as it causes any precipitate. Filter from the precipitated carbonates, and divide the clear filtrate into five equal portions. Reserve one of these portions in case of accident and a second for the tests for organic acid-radicles, and acidify the others whilst they are hot by addition of HCl, HNO_3, and $H\bar{A}$ respectively: examine them as is directed below, using a separate part for each test.

Some metals cannot be completely precipitated as carbonates by boiling with Na_2CO_3 solution: the presence of these metals in solution is, however, frequently of no consequence. If they must be precipitated, addition of Am_2S, or passage of H_2S, will usually separate them as insoluble sulphides, but the subsequent separation of the excess of Am_2S or H_2S by gently warming the solution is troublesome.

Since the analyst knows at this stage of the analysis what metals are present, it should not be difficult to separate them or to allow for their presence while trying the tests for acid-radicles.

The clear acidified portions of the filtrate obtained after boiling with sodium carbonate solution are tested by (443), and the further special tests (446–454) are then tried if necessary.

443.

I. *Portion acidified with HCl.*	*Present.*
On addition of $BaCl_2$ solution a white precipitate insoluble on boiling (444, 1)	$(SO_4)''$
On addition of $BaCl_2$ solution, a semi-transparent precipitate insoluble on boiling (444, 2)	$(SiF_6)''$
On addition of AmCl and Am_2CO_3, a semi-transparent precipitate	$(SiO_3)''$
On addition of $FeSO_4$ solution, a dark blue precipitate	$(Fe_2Cy_{12})^{vi}$
On addition of Fe_2Cl_6 solution, a dark blue precipitate: $FeSO_4$ yielding a light blue precipitate	$(FeCy_6)^{iv}$
On addition of Fe_2Cl_6 solution, a red coloration, destroyed by pouring into $HgCl_2$ solution	$(CyS)'$

II. *Portion acidified with* HNO_3. Refer to (444, 3).	*Present.*
On addition of $AgNO_3$, a *pure white* precipitate, easily soluble in AmHO (444, 1)	Cl′
On addition of $AgNO_3$, a *light yellow* precipitate, with difficulty soluble in AmHO	Br′
On addition of $AgNO_3$, a *yellow* precipitate, almost insoluble in AmHO	I′
Refer to (445, 1).	

III. *Portion acidified with* $H\bar{A}$	*Present.*
On addition of $Pb\bar{A}_2$ solution a yellow precipitate (445, 2).	$(CrO_4)''$
On addition of $CaCl_2$ solution a white gelatinous precipitate	F′*, probably.
On addition of $CaCl_2$ a white pulverulent precipitate	$(C_2O_4)''$*, probably.
On addition of Fe_2Cl_6 a yellowish white precipitate (449, 450).	$(PO_4)'''$, or $(AsO_4)'''$.

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* F will be readily detected in this precipitate, or better in the original substance by (452), $(C_2O_4)''$ by (453).

NOTES ON THE PRECEDING TABLE.

444. 1. Unless the Na_2CO_3 solution used in preparing the solution for these tests was pure, $(SO_4)''$ and $(Cl)'$, if detected, may have been present only as impurities in the Na_2CO_3; portions of the original substance should then be tested by (446) and (447).

2. The presence of $(SiF_6)''$ should be confirmed by adding KCl (300), or by evolving HF by strong H_2SO_4 (301); the HF is most readily obtained from the $BaSiF_6$ precipitate.

3. Should $AgNO_3$ yield a black precipitate, this proves the presence of a sulphide, or possibly of a thiosulphate; add HNO_3 and boil, the black Ag_2S will thus be decomposed, leaving a milky liquid, in which any other precipitate is readily seen after being coagulated by heating or shaking.

445. 1. It must be remembered that $(Cy)'$, $(FeCy_6)^{iv}$, $(Fe_2Cy_{12})^{vi}$, and (CyS) are also precipitated by $AgNO_3$, and therefore if these acid-radicles have been already found, a precipitate produced by $AgNO_3$ does not prove the presence of $(Cl),'$ $(Br)'$, or $(I)'$, which acid-radicles must be specially examined for, as is directed below.

If chloride, bromide, and iodide have all to be tested for, a portion of the Na_2CO_3 solution must be examined by (273); or the precipitate obtained by $AgNO_3$ (443, II.) may be tested by (273 *a*) for Cl, Br, I. If only bromide and iodide have to be tested for, use (274).

2. If a white precipitate of $PbSO_4$ is produced here, it may be dissolved by warming after the addition of AmHO in excess, which will form $Am\bar{A}$ (151); red basic lead chromate will remain if a chromate was present.

SPECIAL TESTS FOR ACID-RADICLES.

446. *Sulphate.*—A portion of the original substance is boiled with HCl, and the decanted or filtered liquid is tested with $BaCl_2$: a white precipitate shows the *presence of* SO_4.

447. *Chloride.*—A portion of the original substance is warmed with HNO_3, the solution decanted or filtered, and $AgNO_3$ added to it; a *perfectly white* precipitate, easily soluble in warm AmHO, shows the *presence of chloride.*

448. *Cyanide* if present will have been detected by its special test (381) and by the smell of bitter almonds, which is given off by the substance after adding H_2SO_4 (411). Ascertain in what form cyanogen is present by (483).

449. *Arsenate.*—This acid-radicle cannot be present unless As was detected during the examination for metals; the presence of $(AsO_4)'''$ is there rendered probable by the precipitation of yellow As_2S_3 occurring only when the acid liquid saturated with H_2S is boiled. If As has been found amongst the metals, proceed to test for $(AsO_4)'''$ by adding

HCl to a portion of the Na_2CO_3 solution (442) until it is acid, then AmCl, AmHO in excess, and $MgSO_4$; filter off any precipitate which forms on warming and shaking the liquid, and pour a few drops of $AgNO_3$ solution upon the white precipitate on the filter, a change of colour to brown shows the *presence of* $(AsO_4)'''$.

450. *Phosphate.*—Boil some of the original substance with dilute HNO_3, and add a little of the clear solution to some $AmHMoO_4$ solution; shake and stir the liquid well, and if no precipitate forms warm *very gently*, a yellow precipitate shows the *presence of* $(PO_4)'''$.

If $(AsO_4)'''$ has been detected (449) this test for $(PO_4)'''$ is only trustworthy when the yellow precipitate has been obtained either in the cold or by employing a *very gentle* heat. If any doubt is felt concerning the presence of $(PO_4)'''$, boil some of the substance with strong HCl, and examine for $(PO_4)'''$ after having entirely separated the $(AsO_4)'''$ by passing H_2S into the boiling HCl solution. A doubtful precipitate with $AmHMoO_4$ may be proved to contain phosphate by dissolving it in AmHO, adding AmCl and $MgSO_4$, filtering and dropping $AgNO_3$ upon the precipitate, if phosphate is present it turns *yellow*, if arsenate *brown.*

451. *Borate.*—Warm a portion of the substance with a little dilute HCl, dip into the solution a strip of turmeric paper and dry it in a steam-oven or at a gentle heat, if the slip is *reddish brown* and becomes *blue black* when moistened with AmHO, *the presence of* $(BO_3)'''$ is shown.

452. *Fluoride.*—Pour upon a portion of the powdered substance strong H_2SO_4; warm the mixture in a leaden or platinum crucible covered with a watch glass, which has been coated with paraffin wax, and has then had characters traced through the film with the point of a penknife: the characters are etched upon the glass (296), showing *the presence of F.*

If SiO_2 is known to be present, the test must be made by conducting the gas, which is evolved when the substance is heated with strong H_2SO_4, into dilute AmHO, when a deposit of gelatinous H_4SiO_4 proves the *presence of* F.

453. *Oxalate.*—The precipitate produced by $CaCl_2$ in III (443), or the original substance if it contains no carbonate, is mixed on a watch glass with MnO_2 free from carbonate, and with strong H_2SO_4, and gently warmed: CO_2 is evolved and detected by holding over the bubbles which rise from the mixture a rod moistened with lime water, or by inverting another watch glass containing on its under surface a drop of lime water over the glass containing the mixture.

453 *a. Tartrate.*—The presence of $\bar{T}$ will probably have been indicated in the Preliminary Examination (388, 412·9). In order to confirm its presence, any metals of Groups I., II. and III. must be separated by H_2S or Am_2S; $Ca\bar{T}$ is then precipitated from the liquid, made just alkaline with AmHO and mixed with a little AmCl, by adding excess of $CaCl_2$, shaking well and letting stand for some time. A precipitate may consist of $Ca\bar{T}$, $Ca_3(PO_4)_2$, or $Ca\bar{O}$. By shaking this precipitate with cold KHO solution, $Ca\bar{T}$ is dissolved and may be reprecipitated from the solution or filtrate by diluting and boiling for some time: the liquid is decanted and the precipitate is then gently warmed with a little *very dilute* AmHO and a crystal of $AgNO_3$, a metallic mirror shows the *presence of* $\bar{T}$.

454. *Sulphide.*—Very small quantities of S may be detected by boiling the substance with KHO for some time, and adding to the clear solution alkaline $Pb\bar{A}_2$ solution, when a black precipitate or coloration will appear.

This concludes the ordinary method of examining a soluble substance: the remaining paragraphs of this section treat of special processes which are necessary for the analysis of certain substances.

REMARKS ON THE PRECIPITATION OF GROUP III.

459. In the General Table (419) it is assumed that in the absence of $(PO_4)'''$ the two Sub-groups III.A. and III.B. can be separated from one another by adding first AmCl and then excess of AmHO to the solution, Group III.A. alone being thus precipitated, and Group III.B. being afterwards precipitated by adding Am_2S to the filtrate.

It is true that AmCl entirely prevents the precipitation of Group III.B. by AmHO if certain other metals are absent and the solution is kept covered from the air; but the members of Group III.A., if they are present at the same time in the solution, are precipitated by AmHO; and the presence of AmCl will not prevent Mn and Zn from being partially precipitated with them, Mn particularly showing a tendency to be precipitated with Fe, and Zn with Cr.

Hence if Mn or Zn be present in small quantity only, it may be entirely precipitated in Group III.A. This is not a serious matter in the case of Mn, since it is readily detected in the ordinary examination of the precipitate by Table III.A. by yielding a *green* mass on fusion with Na_2CO_3 and KNO_3; but Zn may be entirely passed over, since if it had been precipitated with $Cr_2H_6O_6$ its presence would certainly not be detected in Table III.A.

The method of dissolving the precipitate obtained in Group III.A. several times in HCl and reprecipitating it with AmHO, to some extent meets this difficulty; but it is preferable whenever a precipitate is produced on adding AmCl and AmHO, and traces of Mn and more particularly of Zn have to be tested for, to precipitate Groups III.A. and III.B. together, by adding in succession AmCl, AmHO, and Am_2S, and boiling. The precipitate is then examined by Table III.C. (464, 465) for Groups III.A. and III.B., the filtrate being examined, as is directed in the General Table, for Groups IV. and V.

It must be understood that if no precipitate is produced by AmCl and AmHO, Am_2S may be added at once; any precipitate which is formed is then examined by Table III.B., since in the absence of Group III.A. the members of Group III.B. are not precipitated by AmHO in the presence of AmCl.

460. *The presence of* $(PO_4)'''$ *in the HCl solution* which has to be examined for Groups III., IV. and V. involves no special procedure if AmHO added after AmCl produces no precipitate, since the phosphates of Groups III. and IV. and of Mg must be absent. If, however, on adding AmCl and AmHO a precipitate is formed, the directions given at the head of III.D. (466) must be followed, and the precipitate must be examined by that Table.

The reason for this departure from the ordinary course of analysis is that, whereas Al, Cr, Ba, Sr, Ca and Mg, if present as phosphates, are completely precipitated by AmHO, the phosphates of Ni, Co, Mn, Zn and Fe, are only partially precipitated by AmHO; their metals are however entirely precipitated by Am_2S. The filtrate from Am_2S is then examined as is directed in the General Table for Groups IV. and V.

The principles on which the method drawn out in Table III.D. is founded are:—

1. The insolubility of the phosphates of Al, Fe and Cr in $H\bar{A}$ in the presence of an alkaline acetate, the other portions of the precipitate being soluble.

2. The separation of all the $(PO_4)'''$, which is in the $H\bar{A}$ solution combined with Ba, Sr, Ca, or Mg, by the addition of Fe_2Cl_6 in an acetic acid solution.

The further separation of Al, Fe and Cr phosphates is somewhat complicated by the fact that $AlPO_4$ is only decomposed by fusion mixture when SiO_2 is also present, and this SiO_2 has to be removed after the fusion by methods which will be intelligible on reference to (292, 291).

461. In obtaining the precipitate for Table III.D., the precipitates produced by AmHO and Am_2S must be filtered and washed separately; since phosphates of Fe, Zn, Mn, Ni, and Co are converted by Am_2S into sulphides, with formation of ammonium phosphate in solution, and this would precipitate Ba, Sr, Ca, Mg as phosphates even if they were not originally present in that condition. This precipitation would not only complicate the process of analysis, but would also render it impossible to state whether Ba, Sr, Ca and Mg were originally present as phosphates or not.

The phosphates of Groups III. and IV. and of Mg are accordingly first precipitated by AmCl and AmHO, then any members of Group III.B. and any remaining traces of phosphates of Group III. are precipitated from the filtrate by Am_2S. The two precipitates are mixed and treated with Am_2S, which will dissolve away the $(PO_4)'''$ from the phosphates of Fe, Zn, Mn, Ni, Co, leaving the other phosphates undecomposed; hence, on filtering and testing the filtrate with AmCl, AmHO and $MgSO_4$ the formation of a white crystalline precipitate indicates the presence of $(PO_4)'''$ and indirectly establishes the presence in the original precipitate of phosphate of some one or more of the metals Fe, Zn, Mn, Ni, Co.

462. Oxalates, borates, fluorides, and silicates of Ba, Sr, Ca, Mg would likewise be precipitated by AmHO in Group III.A.; but by the evaporation of the HCl solution after passing H_2S, H_3BO_3 and HF are usually volatilised, and H_2SiO_3 becomes insoluble; oxalates are also decomposed by a gentle ignition of the solid substance remaining after evaporation.

The best course to be pursued in precipitating and examining Group III. will be found in (463).

RULES FOR THE PRECIPITATION AND EXAMINATION OF GROUPS III.A. AND III.B.

Refer to (459–462) for the explanation of these Rules.

463. The following rules may be laid down for precipitating and detecting members of Groups III.A. and III.B. in the HCl solution, a small portion of which has been tested for $(PO_4)'''$ by $AmHMoO_4$ (419):—

I. *If the addition of AmHO after AmCl causes no precipitate,* showing the absence of Al, Fe, Cr and of their phosphates, and of phosphates of Ba, Sr, Ca and Mg, Am_2S is at once added, the liquid boiled, and any precipitate which forms is examined for members of Group III.B. by Table III.B. (437): this course is pursued whether $(PO_4)'''$ is present or absent, since even if phosphate is present, metals of Groups III. and IV. and Mg are not present as phosphates.

II. *If the addition of AmHO after AmCl causes a precipitate, showing the presence of Al, Fe, Cr, and $(PO_4)'''$ is not present,* the plan of precipitation depends upon the object of the analyst.

If small quantities of Zn need not be tested for, the method given in the General Table (419) may be followed.

If traces of Zn have to be tested for in the solution, add AmCl, then AmHO and Am_2S in excess, boil, filter, and examine the precipitate by Table III.c_1 (464) or III.c_2 (465). The method described in Table III.c_1 gives trustworthy results, and is to be used for very careful analysis; the method in Table III.c_2 is however much more simple, and is sufficiently accurate for general use.

III. *If the addition of AmHO after AmCl causes a precipitate and $(PO_4)'''$ is present,* the method of precipitation to be adopted together with the Table for the examination of the Group precipitate, will be found in (466)

464. TABLE III.c$_1$.—THE SEPARATION OF GROUPS III.A AND III.B BY $BaCO_3$.

The precipitate may contain Fe, Al, Cr, Zn, Mn, Ni, Co. Rinse it off the filter into a porcelain dish, using as little water as possible, add some strong HCl and boil, adding at intervals a small crystal of $KClO_3$ until all is dissolved but a small quantity of yellow sulphur. Evaporate very nearly to dryness, dilute with a little water, and pour, through a filter if necessary, into a small flask. Cool, pour in a small quantity of $BaCO_3$ suspended in water, cork the flask tightly and shake well; repeat the addition of $BaCO_3$ and agitation until the precipitate is distinctly whitened by the excess of $BaCO_3$: then shake well and allow the flask to stand by corked for at least fifteen minutes, occasionally shaking it vigorously. Let the precipitate subside, filter; wash the precipitate with a little cold water allowing the washings to run through into the filtrate, then wash thoroughly, rejecting the washing-water:—

<table>
<tr>
<td colspan="2">1. <i>Precipitate</i> may contain Fe, Al, Cr, and $BaCO_3$; dissolve it in as little boiling HCl as possible; remove Ba from the <i>boiling</i> solution by adding <i>boiling</i> dilute H_2SO_4 gradually, until after allowing the precipitate to settle, a few additional drops of acid cause no further precipitate (Note 1, below); filter, add pure NaHO in excess to the filtrate, boil and filter:</td>
<td colspan="3">2. <i>Filtrate</i> may contain Zn, Mn, Ni, Co, and $BaCl_2$. Remove Ba by adding to the <i>boiling</i> liquid <i>boiling</i> dilute H_2SO_4 gradually, until the last few drops produce no further precipitate in the clear liquid from which the $BaSO_4$ has been allowed to settle; filter; add pure NaHO in excess to the <i>cold</i> filtrate, stir well and filter:</td>
</tr>
<tr>
<td><i>Precipitate:</i> examine for Fe and Cr by column 2, Table III.A (436).</td>
<td><i>Filtrate:</i> examine for Al by Column 1, Table III.A (436)</td>
<td colspan="2"><i>Precipitate</i> may contain Mn, Ni, Co; rinse off the filter into a porcelain dish with as little water as possible, add some strong HCl and boil; evaporate nearly to dryness, add a little strong solution of $Na\bar{A}$ to the liquid, pass H_2S to saturation, filter:</td>
<td rowspan="2"><i>Filtrate:</i> pass H_2S a white precipitate indicates: <i>Presence of Zn.</i></td>
</tr>
<tr>
<td colspan="2"><i>Note</i> 1.—The separation of Ba may be neglected here, the precipitate being at once boiled with NaHO: in this case much white $BaCO_3$ will remain with the Fe_2O_3 after fusion to separate Cr; it will however dissolve with the Fe and cause no complication.</td>
<td><i>Precipitate:</i> examine for Ni and Co according to Table III.B, column 1 (437).</td>
<td><i>Filtrate:</i> add AmHO in excess; a flesh coloured precipitate shows: <i>Presence of Mn.</i></td>
</tr>
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465. TABLE III.c$_2$.—FOR EXAMINATION OF

Remove the precipitate produced by AmCl, AmHO, and Am_2S
pouring in dilute HCl and gently stirring and shaking the liquid:
filter (see Note 1):—

<table>
<tr><td rowspan="3"><i>Residue</i> if black may contain NiS and CoS; examine it as directed in Table III.B, column 1 (437).</td><td colspan="2"><i>Filtrate</i> may contain Al, Fe, Cr, Zn, Mn. Carefully note its
of this observation, examine the liquid by column I. or II.</td></tr>
<tr><td colspan="2">1. <i>The liquid is perfectly colourless: Absence of Cr.</i>
Boil the liquid in the dish for a few minutes until it no longer smells of H_2S, then add a small crystal $KClO_3$ and boil down to a small bulk; cool, add NaHO in excess, stir well and filter:</td></tr>
<tr><td><i>Precipitate</i> may contain Fe, Mn. Dry and fuse it on platinum foil with Na_2CO_3 and KNO_3; a blue green mass shows:
<i>Presence of Mn</i>.
Boil the foil in a porcelain dish with water for some time, and if any undissolved residue is left, decant the liquid, boil the residue with HCl, and add KCyS; a blood red coloration shows:
<i>Presence of Fe</i> (Note 3, 436).</td><td><i>Filtrate</i> may contain Zn, Al. Divide into two equal parts (Note 2).
Into one portion pass H_2S; a white precipitate forms at once:
<i>Presence of Zn</i>.
To the other portion add AmCl in excess, and heat; a colourless flocculent precipitate:
<i>Presence of Al.</i></td></tr>
</table>

Note 1.—A mere milkiness, due to the separation of sulphur, shows the
Note 2.—Al and Zn may also be detected without dividing the filtrate by
colourless flocculent precipitate shows *Presence of Al:* this is filtered off and H_2S

GROUPS III.A. AND III.B., WHEN MIXED.

from the filter by opening the filter out inside a porcelain dish, take out the filter paper, stir well, and if any black residue is left.

<table>
<tr><td colspan="3">colour, which is best seen by pouring it into a white porcelain dish, and according to the result</td></tr>
<tr><td colspan="3">II. <i>The liquid has a violet or bright green colour: Presence of Cr.</i>
Boil the liquid in the dish until H_2S is no longer smelt, drop in a small crystal of $KClO_3$ and boil down nearly to dryness; dilute with a little water, pour into a small flask and add $BaCO_3$ suspended in water gradually whilst constantly shaking the liquid until the excess of $BaCO_3$ whitens the precipitate, cork the flask and allow it to stand for not less than fifteen minutes, occasionally shaking it well; filter, wash first with cold water letting the washings run into the filtrate, then with boiling water rejecting the washings:</td></tr>
<tr><td rowspan="2"><i>Precipitate</i> may contain Fe, Al, Cr; examine it by column 1, Table III.c_1 (464).</td><td colspan="2"><i>Filtrate</i> may contain Zn, Mn; boil, and whilst boiling add <i>boiling</i> dilute H_2SO_4 gradually until the last few drops cause no further precipitate, filter from $BaSO_4$; cool, add pure NaHO in excess, stir well and filter:</td></tr>
<tr><td><i>Precipitate:</i> dry and fuse with Na_2CO_3 and KNO_3 on platinum foil; blue green mass:
<i>Presence of Mn.</i></td><td><i>Filtrate:</i> pass H_2S; white precipitate:
<i>Presence of Zn.</i></td></tr>
</table>

absence of Ni and Co, and does not render filtration necessary.
adding to it HCl gradually until it becomes acid, then AmHO until alkaline, a
passed into the filtrate, a white precipitate shows *Presence of Zn.*

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TABLE III.D.—FOR EXAMINATION

466. If $(PO_4)'''$ is found in the HCl solution in the General Table
on addition of AmCl and excess of AmHO, the liquid containing the
washed well with hot water. To the filtrate Am_2S is added in excess
Groups IV. and V. (419, 431). The two precipitates yielded by
well with a little Am_2S, then filtered, and the residue on the filter
adding $MgSO_4$; if this is present Fe, Zn, Mn, Ni, Co, one or more

The residue left after stirring with Am_2S may contain Ba, Sr,
Cr as hydrates. Remove it from the filter and heat it with dilute
several small crystals of $KClO_3$, and evaporate very nearly to dryness.
$Na\bar{A}$ (552. 84), as long as any precipitate is produced.

Heat gently, and filter while hot:—

Filtrate: Add Fe_2Cl_6 (Note 2) drop by drop as long as a precipitate forms, and until the liquid, after being well stirred or shaken, remains reddish: addition of Fe_2Cl_6 in large excess must be carefully avoided. Warm gently for some time, filter whilst hot, and wash with hot water:			
Filtrate: Add AmCl, AmHO (Note 3), and Am_2S: filter:			*Precipitate* containing $FePO_4$ may be neglected.
Filtrate: Add Am_2CO_3: filter:		*Precipitate:* examine by Table III.C. (464, 465) for Zn, Mn, Ni, Co, also for Al and Cr. Test a portion of the original solution, or the solution of the substance in HCl, for Fe″ and Fe‴ according to note 3 (436).	
Precipitate may contain $BaCO_3$, $SrCO_3$, $CaCO_3$. Examine this precipitate by Table IV. (438). Ba, Sr, Ca, if found in this precipitate, were present in the solution as phosphates.	*Filtrate* may contain Mg; after removing any traces of Ba, Sr, Ca (Table 439 Column I.) add Na_2HPO_4, warm and shake well; white crystalline precipitate: *Presence of Mg as phosphate.*		

Note 1.—An insoluble residue here may contain $SrSO_4$ and $BaSO_4$ and must
sent originally as phosphates.

Note 2.—A few drops of Fe_2Cl_6 may be added to a small part only of the
at once for Groups III. and IV. and for Mg, without adding Fe_2Cl_6.

Note 3.—Since this filtrate has to be tested for Groups III.A and III.B, it
analysing the precipitate by Table III.C, or by precipitating Groups III.A
precipitates by Tables III.A and III.B (436, 437).

Note 4.—Since $CrPO_4$ is rarely present, this precipitate may generally be tested
detected by adding AmCl in excess which gives a gelatinous precipitate;
adding KCyS.

OF PHOSPHATES IN GROUP III.

(419) after precipitating Groups I. and II., and a precipitate forms
precipitate is gently heated, filtered quickly, and the precipitate is
and the solution boiled, then filtered, and the filtrate examined for
AmHO and by Am_2S are transferred to a porcelain dish and stirred
is washed well. The filtrate should be examined for $(PO_4)'''$ by
were present as phosphates.

Ca, Mg as phosphates; Fe, Zn, Mn, Ni, Co as sulphides; and Al,
HCl in a porcelain dish. If this does not dissolve it, drop in
Filter off S if necessary (Note 1); then add a solution of $H\bar{A}$ and

<table>
<tr><td colspan="4"><i>Precipitate</i> may consist of $FePO_4$ $AlPO_4$, $CrPO_4$ (Note 4). Dry the precipitate on the filter, and fuse it for a short time in a platinum crucible or on platinum foil with a mixture of finely powdered SiO_2, Na_2CO_3, and KNO_3. When cold dissolve by boiling with a little distilled water, add Am_2CO_3; allow to stand for a short time, stirring occasionally; let the precipitate subside, filter:</td></tr>
<tr><td rowspan="3"><i>Filtrate:</i> add $H\bar{A}$ until the solution is acid, boil for a short time, then add $Pb\bar{A}_2$; yellow precipitate of $PbCrO_4$: <i>Presence of Cr, as phosphate.</i>
<i>Note.</i>—A white precipitate on addition of $Pb\bar{A}_2$ may be disregarded.</td><td colspan="3"><i>Precipitate</i> may contain H_4SiO_4, Fe and Al as silicates, and Fe_2Ho_6. Acidify with HCl, evaporate to dryness, and heat the dry residue gently. Warm with a few drops of strong HCl, add hot water, and filter:</td></tr>
<tr><td colspan="2"><i>Filtrate:</i> add pure NaHO in excess, boil and filter:</td><td rowspan="2"><i>Residue</i> is SiO_2 and may be neglected.</td></tr>
<tr><td><i>Filtrate:</i> add AmCl in excess, boil: white, gelatinous precipitate:
<i>Presence of Al as phosphate.</i></td><td><i>Precipitate</i> is brown Fe_2Ho_6: dissolve by boiling with HCl, and add KCyS: blood-red coloration: <i>Presence of Fe as phosphate.</i></td></tr>
</table>

be examined by the latter part of (428): Ba or Sr, if found here, were pre-

filtrate and heated; if it causes no precipitate the other portion may be examined

may be examined either by adding AmCl, AmHO, and Am_2S together and
and III.B separately by AmCl and AmHO and by Am_2S, and examining the

by boiling with excess of NaHO, and filtering; $AlPO_4$ will be in solution, and is
Fe_2Ho_6 remains as a precipitate and is detected by dissolving in HCl and

ANALYSIS OF METALS AND ALLOYS.

467. The metal is first reduced to powder, filings, or thin turnings or shavings. After making the following Preliminary Examination (468), either of the two methods described in (470, 471) may be adopted.

The *first method* should be employed for the examination of a metal or alloy of unknown composition, since all metals, even when present in small quantity only, may thus be detected.

In making the solution by the *second method*, Sn, Sb, Au, and Pt are left undissolved, all other metals passing into solution; it is not however to be recommended as a *general* process for examination of metals and alloys, since the following complications are apt to occur.

If As is present with Sn, a part or the whole of the As may remain in the residue; Pt, if present with a sufficient quantity of Ag, may pass entirely into solution; and Sb will always partly dissolve. The process however is useful in cases where an alloy, known to contain a large proportion of Sn or Sb, has to be examined for other metals, since these are dissolved away at once from the bulk of the Sn or Sb, but care must be taken that As is not passed over in the presence of Sn.

PRELIMINARY EXAMINATION.

468. Note the colour of the substance, and also any smell that may be given off when it is rubbed with the hand; also whether it is crystalline or not, and if it is attracted by a magnet (Fe, Ni, Co, &c.).

Note also the hardness of the metal, by seeing if it can be scratched or cut by a steel knife; also whether on being struck smartly with a hammer it breaks to powder (brittle), or flattens out (malleable): then try the following experiments with separate small portions of the substance:—

I. Heat with Na_2CO_3 on charcoal in the inner blowpipe flame (403, 404, 405).

II. Fuse into a colourless borax bead (401).

III. Try the flame coloration (397).

IV. Heat in an ignition tube, and note whether a metallic sublimate of Cd, As, or of globules of Hg forms; or whether S sublimes from a sulphide.

469. P, As and S can frequently only be detected as phosphate, arsenate and sulphate by dissolving the substance in strong HNO_3, or in aqua regia, or by fusing it with KNO_3 and Na_2CO_3 and dissolving in water, and testing the solution by (450, 449, 446).

Si can be detected as SiO_2 by evaporating the acid solution (428).

Solution and Examination of a Metal or Alloy.

470. Method I.—Pour some rather dilute HCl upon the powdered metal in a small flask covered with a watch-glass with its concave surface upwards, and heat for some time just short of boiling; if the metal dissolves readily, continue heating until the metal is completely dissolved, and examine the HCl solution according to the General Table (419).

Frequently HCl alone does not effect complete solution, two or three drops of strong HNO_3 should then be poured in, and more HNO_3 and HCl added occasionally when the action ceases or when red fumes are no longer given off on heating. When the metal has entirely disappeared, add a little more strong HCl and boil as long as any Cl or reddish fumes are given off; then dilute with a little water, heat to boiling, and cool; filter if there is any white residue :—

Residue, if crystalline, is probably $PbCl_2$, and will be found to dissolve entirely if washed with sufficient boiling water. In this solution the presence of Pb is confirmed by adding K_2CrO_4, which gives a yellow precipitate soluble in excess of KHO, showing *Presence of Pb.* If any residue is left after washing well with boiling water, it is probably AgCl; confirm the presence of Ag by pouring upon the residue hot AmHO, it dissolves completely and is reprecipitated on adding HNO_3 in excess :— *Presence of Ag.*	*Filtrate* (note 1): dilute largely with H_2O (see note 2), and whether this causes a precipitate or not, pass H_2S to saturation into the cold solution; examine any precipitate thus produced by Table II. (435), and proceed to examine the filtrate for Groups III., IV., and V., as directed in the General Table (419). *Note* 1.—If Au and Pt may be present, they must be tested for in the H_2S precipitate, according to the directions in (472–474). *Note* 2.—A white precipitate appearing on dilution is due to the presence of Bi, Sb, or Sn.

471. METHOD II.—Pour upon the finely divided metal some strong HNO_3*, and heat in a small flask covered with a watch glass, as long as any red fumes appear: one of two results will occur:—

I.	II.	
The substance dissolves completely with or without addition of water. Absence of Pt, Au, Sb, Sn (note 1). Examine the solution, after boiling nearly to dryness and diluting with water (note 2), by the General Table (419).	*A residue is left:* add some hot water and boil, then filter and wash the residue on the filter well with boiling water (note 2). The residue may present the following appearances:—	
	1. *Entirely metallic or black powder:* probably Pt or Au. Dissolve by heating in a small flask covered with a watch glass, with a little HCl to which a few drops of HNO_3 have been added. When completely dissolved add more HCl, and boil down in an evaporating basin nearly to dryness, examine the solution for Au and Pt by (474), commencing with the addition of KCl and using only the left hand side of the table, since Sn will be absent.	2. *White powder* may contain Sn, Sb, As, possibly also Pt and Au concealed in it (note 3).

Note 1.—The solution is liable to contain small quantities of these metals, which must always be tested for in a careful analysis.

Note 2.—BiOCl will often precipitate on dilution, but the precipitate will disappear on adding HCl and boiling, being thus easily distinguished from the Group I. precipitate.

Note 3.—If much residue is obtained a small quantity of it may be heated in a test-tube with HCl adding $KClO_3$, if it dissolves entirely dissolve the whole of the residue, then examine the solution by Table II., commencing at (435 *b*): if it refuses to dissolve proceed with the rest of the residue as directed below. If the residue is small in quantity examine it at once as directed below.

Examination of the Non-metallic Residue insoluble in HNO_3.—Dry the residue on the filter at a gentle heat, mix it thoroughly with about an equal quantity of powdered Na_2CO_3 and $NaNO_3$, and add the mixture gradually to some $NaNO_3$ in fusion in a porcelain crucible; then pour the melted substance out into a porcelain dish; allow it to cool then pour upon it cold water and let it stand for some time. After crushing the mass with a pestle and stirring it occasionally, filter, and wash the residue on the filter with dilute alcohol, throwing away the washings:—

---

* If Hg is found in the preliminary examination and small quantities of Ag have to be tested for, the Hg should be expelled by heating the substance strongly in a porcelain crucible before dissolving in acid, as $Hg(NO_3)_2$ tends to prevent the precipitation of Ag by HCl in Group I.

Residue may contain Sn, Sb, Pt, Au. Place it in a small porcelain dish, pour in a little HCl and heat, then add water; whether the residue has dissolved or not place in the liquid a strip of Pt-foil and drop upon it a piece of pure Zn; remove the platinum strip in a few seconds; if it is stained black *Sb is present.* Wait until the evolution of H ceases, taking care that there is some Zn left undissolved, and adding more Zn if the first piece has entirely dissolved. The residue in the dish may consist of Sn, Au, and Pt; remove the Zn, rinsing off any substance adhering to it into the dish, stir the liquid in the dish well, then pour off the liquid, carefully leaving the residue; pour in water, stir well, and again pour off carefully. Boil the residue for some time with strong HCl in a test-tube, dilute, decant, add $HgCl_2$, a white precipitate forms:—*Presence of Sn.* *Residue:* dissolve by warming with HCl and HNO_3, and examine the solution for Au and Pt by (474), using only the left hand portion of the table.	*Filtrate* may contain $(AsO_4)'''$: add HNO_3 until the solution is acid and boil, evaporating the liquid in a dish if very bulky. Pour into half this solution $AgNO_3$ as long as it gives any precipitate, and add gradually AmHO diluted with 10 or 12 times its bulk of water, a brown precipitate shows:— *Presence of As.* To the other half of the acid solution add AmHO in excess, then $MgSO_4$, and rub the inside of the vessel with a glass rod; a white crystalline precipitate, often appearing only after some time, shows:— *Presence of As.*

SEPARATION AND DETECTION OF Au AND Pt.

472. Au and Pt will, in the ordinary course of analysis, be entirely precipitated as sulphides in the second Group, if H_2S is passed for some time into the hot HCl solution; and since these sulphides are soluble in KHO and in Am_2S, on examining the H_2S precipitate by Table II. (435), the Au and Pt will pass into the filtrate when the precipitate is boiled with KHO or Am_2S. No modification of the process described in Table II. is required for the detection of these two metals until the examination of the residue in the hydrogen flask is commenced. The Au and Pt will be present in this residue, associated with Sn if it be present, and usually also with at least a part of the Sb (if present), since Zn in contact with Pt in an acid liquid causes the separation of metallic Sb (193). Accordingly when Au and Pt have to be tested for, the ordinary course of analysis is to be followed until the residue in the hydrogen flask is obtained; and this is examined as directed in (473).

473. After washing the residue left in the hydrogen flask by decantation in a porcelain dish, and removing any excess of Zn, boil it with a little strong HCl in a test-tube for several minutes, allow the residue to settle, and decant the liquid:—

<table>
<tr><td colspan="2">474. <i>Residue</i>*; pour upon the residue in the dish a little HCl, add several drops of HNO_3, and boil gently very nearly to dryness; add some KCl solution and evaporate once more very nearly to dryness. Pour some absolute alcohol into the cool dish and stir well for a time, allow the precipitate to settle and decant the liquid, wash the precipitate by stirring it with a little more alcohol and decant the liquid when the precipitate has settled:—</td><td rowspan="2"><i>Solution:</i> to one part add $HgCl_2$ a white precipitate, which does not appear at once if only traces of Sn are present:
<i>Presence of Sn</i>.
Pour the other part of this solution into a porcelain dish, immerse in it a strip of Pt and drop a piece of Zn on the Pt; a black stain on the Pt shows:—
<i>Presence of Sb</i>.</td></tr>
<tr><td><i>Precipitate</i> will consist of yellow K_2PtCl_6 and excess of KCl, dissolve it in a little boiling water, pour it into a white dish, add several drops of HCl, then $SnCl_2$: an orange red coloration confirms the
<i>Presence of Pt</i>.</td><td><i>Solution</i> will be yellow if Au is present; evaporate carefully on a water-bath just to dryness, dissolve in a little water and add a few drops of freshly prepared $FeSO_4$ solution. Au will be precipitated in a fine powder, causing the liquid to appear reddish by reflected and blue by transmitted light:—
<i>Presence of Au</i>.</td></tr>
</table>

* If Sb has not been detected already in the precipitate produced by the gases in $AgNO_3$ solution (435 *b*) this residue should be examined for Sb, as the Sb instead of having been given off as SbH_3, may have been deposited on the Pt or Au. Boil it once more for some time with strong HCl to remove all Sn, wash the residue well by decantation and boil it in the dish with $H_2\bar{T}$ adding a few drops of HNO_3, decant, and test the liquid for Sb by adding HCl and passing H_2S: the residue is then examined as above (474).

EXAMINATION OF SUBSTANCES INSOLUBLE IN WATER AND ACIDS.

475. *An insoluble substance may consist of* one or more of the following substances, all of which are white except Fe_2O_3, Cr_2O_3, $FeCr_2O_4$, $PbCrO_4$, S, C, native SnO_2 and CaF_2, and AgCl which has been exposed to light.

In the following list those substances which are embraced in brackets ( ) may *possibly* be present, those in square brackets [ ] *improbably* because they are soluble in water.

1. $BaSO_4$	Insoluble in water and acids.
2. $SrSO_4$	Insoluble „ „
3. $[CaSO_4]$	Not perfectly insoluble in water, soluble in hot HCl, and should therefore pass into the acid solution.
4. $(PbSO_4)$	
5. $PbCrO_4$	Insoluble after being strongly heated.
6. $[PbCl_2]$	Soluble in boiling water, and should therefore have been removed if the residue was well washed with boiling water.
7. $AgCl$	This may have been originally present as such, or may have been derived from the use of HCl in making the solution, or by the action of aqua regia on the insoluble substances AgBr, AgI, AgCy, $Ag_6Fe_2Cy_{12}$, Ag_4FeCy_6.
8. SiO_2	Either uncombined, or as a silicate.
9. (Al_2O_3)	Insoluble after being strongly ignited, but usually dissolved by long boiling with strong HCl.
10. (Fe_2O_3)	
11. (Cr_2O_3)	
12. $FeCr_2O_4$	Chrome iron ore, native.
13. (SnO_2)	Native or ignited.
14. Sb_2O_5, Sb_2O_4	
15. CaF_2	Also a few other fluorides, and some metaphosphates and arsenates.
16. S	Yellow, slowly soluble in strong HNO_3, giving red fumes, and yielding H_2SO_4.
17. C	Black, and quite insoluble.

If sufficient of the substance is at disposal, the preliminary examination (476) may be made on a portion of it. In case the quantity of substance is small, however, the whole of the substance must be employed for the examination by fusion (477).

Preliminary Examination of Insoluble Substances.

476. The substance must be in the state of dry powder. Make a careful examination of it with a pocket lens. Expts. I. and II. need only be made if the substance is light in colour.

Experiment.	Observation.	Inference.
I. Observe whether the substance darkens when allowed to stand in the light for some time.	The colour changes to violet or black.	Presence of $AgCl$.
II. Pour a little Am_2S upon a portion of the substance on a watch glass.	The substance blackens; pass on to III.	Presence of Pb or Ag.
	The substance does not blacken; pass on to V., omitting III. and IV., since Pb and Ag must be absent.	
III. Heat some of the substance with a little water and a small piece of KCy; filter off, keeping the residue; to the filtrate add Am_2S.	A brownish precipitate.	Presence of $AgCl$. *Confy.* On warming some of the substance with $AmHO$, filtering, and adding excess of HNO_3 to the filtrate, a white precipitate forms, which, when shaken well or heated, coagulates into flocks.
IV. Wash the residue from Exp. III. well upon the filter:—		
a. It is white; drop Am_2S upon it.	The residue blackens.	Presence of $PbSO_4$ or $PbCl_2$.
b. It is dark coloured: add water and $H_2\bar{T}$, and boil after adding $AmHO$ in excess; filter, to the filtrate add $H\bar{A}$ in excess and K_2CrO_4.	A yellow precipitate, soluble in KHO.	Presence of $PbSO_4$ or $PbCl_2$.
V. Take up some of the substance upon a moistened loop of platinum wire; heat it for a short time in the inner blowpipe flame, then moisten with a drop of strong HCl, and hold in the outer part of a Bunsen flame.	A *reddish yellow* coloration, *dusky green* when seen through the indigo prism. A *crimson red* coloration appearing *deep red* through the indigo prism. A *yellowish green* coloration. These colorations may often be seen in succession and further distinguished by the spectroscope.	Presence of Ca Presence of Sr Presence of Ba } as sulphate.
VI. Heat in a small test-tube or ignition-tube, then strongly on a piece of porcelain or broken glass, or on platinum foil.	A yellow sublimate forms on the sides of tube.	Presence of S.
	When strongly heated the substance smoulders, and ultimately burns away.	Presence of C.

Experiment.	Observation.	Inference.
VII. Heat with strong H_2SO_4 in a platinum crucible or leaden cup covered with a watch glass (296): or if a silicate is present examine by passing the gas into AmHO (297).	The glass is etched.	Presence of F.
	A gelatinous precipitate is obtained in the AmHO.	Presence of F.
VIII. Fuse some of the substance in a bead of $NaAmHPO_4$, first in the outer then in the inner blowpipe flame.	Particles are seen floating undissolved in the melted bead.	Presence of SiO_2 (480).
	Green coloured bead.	Presence of Cr.
	Reddish brown bead, colourless when cold, and becoming *greenish* in the inner flame.	Presence of Fe.

EXAMINATION OF INSOLUBLE SUBSTANCES.

477. Mix the finely powdered substance* with five or six times its bulk of fusion mixture, and heat the mixture in a small covered porcelain crucible† until it melts; keep it in fusion for at least ten minutes. Allow the crucible to cool, then pour some water into it and allow to stand or boil until the solid mass is loosened from the crucible; boil this in a porcelain dish with distilled water, crushing the mass by pressure with a pestle if it does not quickly fall to pieces. Allow the residue to settle, pour off the solution through a filter, and boil the residue with a little more water; pour off through the same filter, adding this filtrate to the former one.

For the examination of the undissolved residue see (478), for the filtrate (479).

478. *Residue on the filter;* wash well with boiling water, then make a hole in the bottom of the filter, wash the residue through into a test-tube by pouring upon it a little boiling dilute HNO_3 (note 1), and boil; the residue dissolves entirely if the fusion has been continued sufficiently long (note 2). Filter if necessary, and examine the solution by the General Table (419), bearing in mind that only the metals enumerated in (475) are likely to be present (note 3).

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* Which, if it is found by Exp. VI. to contain free S or C, must first be heated strongly for some time in an open porcelain crucible.

† If Pb and Ag have been proved to be absent by the preliminary tests, or if they have been first removed by boiling the substance with KCy solution, then with $H_2\bar{T}$ and excess of AmHO and washing well, a platinum crucible may be used. The use of a platinum crucible is preferable, since after fusing in porcelain small quantities of Al_2O_3 and SiO_2 will always be found, being derived from the action of the Na_2CO_3 on the glaze of the porcelain.

Note 1.—If Ag and Pb are known to be absent by the preliminary experiments, HCl may be used for dissolving the residue and is preferable; if effervescence is produced by these acids the presence of Ba, Sr, Ca or Mg is proved.

Note 2.—A residue here may consist of chrome iron ore; this may be dissolved by heating with HNO_3 and $KClO_3$, and the solution tested by the General Table, when Cr and Fe will be found.

Note 3.—It is best to evaporate the solution quite to dryness before testing for Group III., using a water-bath towards the end if the substance spirts. This is always necessary if SiO_2 is present in the substance, since it sometimes passes into the acid solution, and is completely separated from this solution by the process of evaporation (291).

479. *Aqueous solution of the fused mass.*—Divide this solution into two parts A and B.

A. Add HCl until the liquid is distinctly acid, and evaporate in a porcelain dish to dryness, finishing the process on a water-bath if necessary to avoid spirting; then continue to heat the dish gently until the residue is quite dry; pour in a little strong HCl and warm, dilute and heat again; an insoluble residue shows *presence of* SiO_2.

Filter, and examine the filtrate by the General Table (419) more particularly for Al, but other bases, such as Cr, Mn, Zn, Sn, should also be tested for, since they are soluble in alkalis and may therefore pass into this solution.

B. Test separate portions for the following acid-radicles as described below: unless the fusion mixture employed was tolerably free from chloride, sulphate and phosphate, tests 1, 2, and 4 may be of little value.

1. *Chloride:* acidify with HNO_3 and add $AgNO_3$, white precipitate easily soluble in AmHO.
2. *Sulphate:* acidify with HCl and add $BaCl_2$, white precipitate insoluble on boiling.

3. *Chromate:* acidify with $H\bar{A}$ and add $Pb\bar{A}_2$, yellow precipitate; a chromate is also seen by the yellow colour of the solution. Cr thus detected may have been present acting as a metal or as a constituent of an acid-radicle.

4. *Phosphate:* acidify with HNO_3, add a few drops to some $AmHMoO_4$ solution, and warm gently. A yellow precipitate forms, often only after a time, or on gently heating. If As has been found on passing H_2S into the hot HCl solution, $(PO_4)'''$ must be tested for in a portion of the filtrate after boiling off H_2S, else the above yellow precipitate may be due to $(AsO_4)'''$.

5. *Fluoride:* add HCl in excess to a part of the solution, stir well, and let stand until the CO_2 has escaped, then add AmHO in excess, then $CaCl_2$ as long as it produces any precipitate, and let stand for a time; filter off, dry the precipitate and examine it for F by pouring strong H_2SO_4 upon it in a platinum crucible covered with a watch glass (296.)

ANALYSIS OF SILICATES.

480. The presence of silica is shown by (382) or by Exp. VIII. in the Preliminary Examination of insoluble substances (476); when it has been found it becomes necessary to examine for all metals, since many silicates which are soluble when alone, become insoluble when they are mixed or combined with insoluble silicates.

Many silicates are entirely decomposed by heating them with strong HCl for some time just short of boiling; if the decomposition is complete, only a colourless residue of silicic acid will remain, and this may be identified by its solubility in hot Na_2CO_3 solution.

If the silicate is not completely decomposed by hot strong HCl, it is treated as is directed in (477–479), remembering however that all metals may be present.

481. Since Na and K cannot be tested for in the solution obtained after fusion with alkaline carbonates, a separate portion must be examined for these metals by one of the two following processes: the materials used in the tests must be perfectly free from K and Na.

METHOD I.—The finely powdered silicate is mixed with its own weight of sublimed and crystallised NH_4Cl in powder, and with eight times its weight of pure $CaCO_3$ (see below). The mixture is gently heated in a platinum crucible for a few minutes, and finally kept at a bright red heat for twenty or thirty minutes: the mass will not fuse as a whole, but the fused $CaCl_2$ will dissolve sufficient CaO to bring this into contact with the silicates and decompose them.

The cool substance after being turned out of the crucible, if possible, is boiled with water for some time after it has crumbled by the slaking of the CaO: the liquid is then filtered, and Am_2CO_3 solution is added to the filtrate until it causes no further precipitate of $CaCO_3$. The filtrate from this precipitate is evaporated considerably and freed from traces of Ca by the addition of $Am_2C_2O_4$. The clear solution will now contain K, Na, Li, if they were present, as chlorides; they may be tested for by the right hand side of Table V. (439), and by the spectroscope (42).

The pure $CaCO_3$ required for this process is prepared by dissolving marble in HCl, and then adding powdered marble in excess and warming. The solution is then mixed with lime water or milk of lime until it is alkaline in reaction: magnesium, calcium phosphate and iron are thus precipitated. This solution is heated to about 75° C., and warm Am_2CO_3 solution is added until it causes no further precipitation; the precipitated $CaCO_3$ is filtered off and washed well on the filter.

METHOD II.—Evaporate the powdered substance several times in a platinum dish or crucible either with hydrofluoric acid and subsequently with strong H_2SO_4, or with five times its weight of finely powdered calcium fluoride mixed into a paste with strong H_2SO_4; in either case the mass is finally heated until no more white fumes are evolved. The cool residue is then boiled with water, $BaCl_2$ solution is added as long as it causes any precipitate, then AmHO is added in excess and Am_2CO_3 solution is poured in as long as it causes any precipitate; the precipitate is filtered off and the filtrate is examined for K and Na by Table V. (439), and by the spectroscope (42).

ANALYSIS OF SUBSTANCES CONTAINING CYANOGEN.

482. If the substance to be analysed is found to contain cyanogen (381), the usual course of analysis must frequently be somewhat modified, since the presence of cyanogen might produce confusing results.

The cyanogen may be present as a cyanide, sulphocyanide, ferrocyanide, ferricyanide, cobalticyanide, and rarely as a manganocyanide or chromicyanide. It is necessary first to ascertain in what form the cyanogen occurs, by trying the following preliminary experiments on a small portion of the substance.

PRELIMINARY EXAMINATION.

483. Boil a portion of the substance for several minutes with KHO solution, then add some Na_2CO_3 solution as long as it causes any precipitate, and boil again for several minutes; filter, make the cold filtrate just acid with HCl, filter if necessary, and test separate portions as follows:—

Reagent added.	Result.	Inference.
1. $FeSO_4$ solution	Blue precipitate	Presence of $(FeCy_6)^{iv}$ $(FeCy_6)_2^{vi}$.
	White precipitate	Probable presence of $(CoCy_6)_2^{vi}$.
2. Fe_2Cl_6 solution	Blue precipitate	Presence of $(FeCy_6)^{iv}$.
	Blood red coloration	Presence of $(CyS)'$.
3. Add $ZnSO_4$ solution as long as it causes any precipitate.	Light brown precipitate	Presence of $(FeCy_6)_2^{vi}$.
	White precipitate	Presence of $(FeCy_6)^{iv}$, $(CoCy_6)_2^{vi}$.
The precipitate produced by $ZnSO_4$ should be filtered off and fused into a colourless borax bead.	A blue bead is produced	Presence of $(CoCy_6)_2^{vi}$

484. If a *simple cyanide* only is present the ordinary course of analysis is pursued, but it will be necessary to remove HCy by boiling the solution for some time after adding an acid (418, 419).

If a *sulphocyanide* is found by the preliminary tests, it may usually be decomposed in the portion of the substance which is to be examined for metals by pouring strong HNO_3 upon it in a porcelain dish and boiling down nearly to dryness: the liquid is then diluted and boiled, and may be considered as solution III. (418), any undissolved residue being treated as is directed in the Table.

485. Should the preliminary tests have proved the presence of ferro-, ferri-, cobalti-, chromi-, or mangano-cyanide, two methods of procedure are open to the analyst. Either the cyanogen may be removed from the substance before commencing the analysis (486), or the substance may be examined without any such preliminary treatment (487).

The advantage of employing the more complicated method in (487) is that it enables the analyst to decide whether the metals are present in cyanogen acid-radicles or not, and it yields a more precise knowledge of the constitution of the substance.

486. METHOD I.—If cyanogen is not present only as a simple cyanide or sulphocyanide, either of the following two processes may be used for removing it from the substance before analysis; the substance may then be examined for metals in the usual way.

1. Pour upon the powdered substance strong H_2SO_4 in a porcelain crucible, evaporate to dryness and ignite the residue strongly; when cold dissolve it by heating with a little strong HCl, adding water and heating again.

2. Fuse the substance in a porcelain crucible with three or four times its weight of a mixture of 3 parts of Am_2SO_4 and 1 part of $AmNO_3$: the residue left in the crucible will be free from cyanogen and may be subjected to ordinary analysis.

These lines run across both pages.

487. Method II. (Fresenius).—Boil the substance with water, to the filtrate :—

<table>
<tr>
<td rowspan="5"><i>Filtrate:</i> examine this for metals, more especially the alkalis, and for acid-radicles according to the directions given for analysing a liquid (383).
The acid-radicles should be first tested for in a portion of the solution and if $(FeCy_6)^{iv}$, $(Fe_2Cy_{12})^{vi}$, $(Co_2Cy_{12})^{vi}$, one or more be found, the solution must be evaporated to dryness with HNO_3, and the residue strongly heated (see General Table) after filtering from the H_2S precipitate, in order to destroy these cyanogen radicles. The Fe, Co, &c., in the cyanogen acid-radicles will then be detected in the General Table.</td>
<td colspan="3"><i>Residue:</i> boil with KHO solution for several minutes, then add and boil again ; filter and wash the residue :—</td>
</tr>
<tr>
<td colspan="3"><i>Filtrate:</i> pass H_2S, and if it causes any precipitate continue KHO*, heat and filter :—</td>
</tr>
<tr>
<td colspan="2" rowspan="2"><i>Precipitate:</i> wash well with boiling water, and boil the precipitate with strong HNO_3, a black residue of HgS may remain, filter this off after diluting the acid, and confirm the presence of Hg in the precipitate by heating it with Na_2CO_3 in a bulb-tube (147).
The filtrate (or solution if HgS is absent) is evaporated to dryness, the residue is then dissolved in a little hot strong HCl, the solution is much diluted, saturated with H_2S, and filtered :—</td>
<td><i>Filtrate:</i> add dilute saturation and filter :—</td>
</tr>
<tr>
<td rowspan="2"><i>Precipitate:</i> boil with KHO, filter off any black precipitate and examine it for Hg by heating it with Na_2CO_3 in a bulb-tube (147).
Add to the filtrate or solution HCl until it is acid, pass H_2S to saturation, and examine for As, Sb, Sn, (Pt, Au), by Table II. (435), commencing at (435 <i>b</i>).</td>
</tr>
<tr>
<td><i>Precipitate:</i> examine for Pb and Cu by Table II. (435 <i>a</i>).</td>
<td><i>Filtrate:</i> add AmCl, AmHO in excess, and Am_2S, and examine for Zn, Mn, Ni, Co, &c., by Table III.c_2 (465).</td>
</tr>
</table>

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* If H_2S causes a precipitate, NaHS or KHS may be added, drop by filtrate; this may be done instead of passing H_2S to saturation and then

filter, and wash the residue with boiling water, adding the washings

<table>
<tr><td colspan="4">some Na_2CO_3 solution as long as it causes any precipitate in the solution or the filtrate,</td></tr>
<tr><td colspan="3">to pass the gas until the liquid is saturated, then add more</td><td rowspan="5"><i>Residue:</i> dissolve and examine for metals in the usual manner (418).
Fe, Co, Mn, Cr, if found in this residue, were not present in cyanogen acid-radicles.</td></tr>
<tr><td colspan="3">HNO_3 gradually until the liquid is just acid, pass H_2S to</td></tr>
<tr><td colspan="3"><i>Filtrate:</i> Divide into two parts, α, β:—</td></tr>
<tr><td rowspan="2">α. Examine for acid radicles in the usual way, testing for $(Co_2Cy_{12})^{vi}$ by adding excess of $ZnSO_4$, filtering and trying whether the precipitate gives a blue bead with borax.</td><td colspan="2">β. Evaporate to dryness and fuse the residue: when cold boil it with water and filter:—</td></tr>
<tr><td><i>Residue:</i> dissolve in HCl, and test for Al, Fe, Mn, Co (465); the last three if found were present as cyanogen acid-radicles.</td><td><i>Filtrate:</i> acidify a portion if yellow, with $H\bar{A}$ and add $Pb\bar{A}_2$, a yellow precipitate shows $(CrO_4)''$, the Cr having been present as cyanogen acid-radicle.
Test another part for Al by adding HCl in excess, then AmHO in excess.</td></tr>
</table>

drop, until it no longer gives any precipitate in the liquid or in the

adding KHO.

EXAMPLE SHOWING HOW TO ENTER THE RESULTS OF ANALYSIS OF A COMPLEX SUBSTANCE.

488. The substance given for analysis consisted of a powder containing pink, blue, white, and black particles. It smelt faintly of ammonia.

PRELIMINARY EXAMINATION FOR METALS.

Experiment.	Observation.	Inference.
EXPT. I.—Heated in a small dry test-tube.	Water given off which turned red litmus-paper blue.	Pres. of H_2O. Pres. of NH_4.
	Strong smell of NH_3 gas.	Pres. of NH_4.
	Substance blackened, no smell of burning.	Prob. pres of Co, Cu, and abs. of $\bar{T}$ and $\bar{A}$.
	Slight white sublimate.	Pres. of As, NH_4, Hg.
Confy.—Held in the upper part of the tube a glass rod with a drop of lime water hanging on its end.	The lime water became milky.	CO_2 evolved.
	Brown nitrous fumes evolved, recognised by their smell.	Pres. of nitrate.
	Cl-gas evolved, found by smell and bleaching litmus.	Pres. of Cl.
Confy.—Boiled a portion of the substance with KHO solution.	NH_3 gas was evolved, recognised by its smell. and by giving white fumes with strong HCl.	Pres. of NH_4.
Confy.—Heated strongly another portion of the dried substance with Na_2CO_3 in a bulb-tube.	No mirror formed.	Abs. of Hg and As.
EXP. II.—Heated a portion of the substance on a loop of platinum wire in the Bunsen flame, moistened with HCl and heated again in the flame.	*Bright yellow* flame. The flame appeared *crimson* through the indigo-prism.	Pres. of Na. Pres. of K.
Heated for some time in the tip of the inner blowpipe flame, moistened with HCl, and again held in the Bunsen flame	*Crimson* col^{n}. appearing *intense red* through the indigo-prism. *Bright green* col^{n}. with *blue* core.	Pres. of Sr. Pres. of Cu.

Expt.	Observation.	Inference.
Expt. III.—Heated a portion of the substance on charcoal in the inner blowpipe flame.	The greater part of the substance fused readily, and was absorbed by the charcoal.	Pres. of a salt of K, Na.
	Deflagration occurred.	Pres. of chlorate, nitrate.
	A red metallic residue remained.	Pres. of Cu.
Confy.—Fused in a clear borax bead in the outer and inner blowpipe flames.	In the outer flame a bead *green* whilst hot, *blue* when cold. In the inner flame *red and nearly opaque.*	Pres. of Cu
Confy.—Fused on platinum foil with Na_2CO_3 and KNO_3.	No *bluish green* or *yellow* mass on cooling.	Abs. of Mn and Cr.
Confy.—Fused on charcoal in the inner blowpipe flame with Na_2CO_3.	Red metallic residue.	Pres. of Cu.
	A portion placed on a silver coin and moistened gave no black stain.	Abs. of S.

Preliminary Examination for Acid-radicles.

Expt.	Observation.	Inference.
Expt. I.—Added dilute HCl without heating.	A colourless gas was evolved, which was free from smell, and turned a drop of lime water milky.	Pres. of carbonate. Abs. of sulphite, hypochlorite, &c.
Heated to boiling.	Cl was evolved, recognised by its smell and by bleaching moist litmus-paper.	Pres. of nitrate, chlorate, or some other oxidising substance.
Expt. II.—Added strong H_2SO_4.	A bright yellow chlorous gas evolved, which crackled when warmed.	Pres. of chlorate.
Confy. — Dropped in copper turnings and heated.	Reddish brown fumes evolved.	Pres. of nitrate.
Heated strongly, cooled, and rinsed out.	The tube when dry was seen not to be etched.	Abs. of fluoride.
Confy.—Boiled a portion of the substance with water, added strong H_2SO_4, cooled and poured in $FeSO_4$ solution carefully.	A brown ring formed on the surface of the acid.	Pres. of nitrate.

Examination for Metals in the Wet Way.

Boiled a portion of the substance with Fe_2Cl_6, $FeSO_4$ and KHO, added HCl in excess, no blue pp.:—*Absence of Cy.*

These lines run across both pages.

Boiled a portion of the substance with water; as it did not upon the undissolved residue : effervescence occurred, and on boili Cl was perceived; the substance was completely dissolved. C previously to give no pp. on addition of a little dilute HNO_3 :—

<table>
<tr><td rowspan="3"><i>No pp.</i>
<i>Abs. of Group I.</i>
Hg′, Ag, and prob. Pb.</td><td colspan="2">Diluted with water and passed H_2S until the liquid smelt s</td></tr>
<tr><td rowspan="2"><i>A black pp.</i>
Exam$^{d.}$ by Table II.</td><td><i>Filtrate</i> which gave no further p Boiled until it no longer smelt on addition of HNO_3, (<i>prob. pres</i> of the HCl sol$^{n.}$ to some Am boiled; filtered :—</td></tr>
<tr><td><i>A brown pp.</i>
Exam$^{d.}$ by Table III.A.</td></tr>
</table>

EXAM$^{N.}$ OF PP. IN GROUP II.—Removed from the filter into a porcelain dish and boiled with KHO, filtered :—

<table>
<tr><td rowspan="4"><i>Filtrate</i> :— Acidified with HCl, a <i>white</i> milky liquid only: <i>Abs. of Group</i> II. B.</td><td colspan="4"><i>Pp.</i> removed from <i>filter</i> by a glass rod into a porcelain dish and boiled with strong HNO_3 as long as any red fumes came off, added dilute H_2SO_4 and stirred well:</td></tr>
<tr><td rowspan="3"><i>No pp.</i> :—
<i>Abs. of Hg and Pb.</i></td><td colspan="3">Added to sol$^{n.}$ excess of AmHO: blue solution (<i>Pres. of Cu</i>):—</td></tr>
<tr><td rowspan="2"><i>No pp.</i> :—
<i>Abs. of Bi.</i></td><td colspan="2">Acidified the blue sol$^{n.}$ with HCl and saturated with H_2S; filtered off the black pp. rapidly and boiled it with dilute H_2SO_4:</td></tr>
<tr><td><i>Pp.</i>: dissolved in a little boiling dilute HNO_3, added AmHO in excess, then excess of $H\bar{A}$, then K_4FeCy_6, chocolate red pp. :—
<i>Pres. of Cu.</i></td><td><i>Filtrate</i>: diluted much and passed H_2S, no pp. :—
<i>Abs. of Cd.</i></td></tr>
</table>

EXAM$^{N.}$ OF PP. IN GROUP IIIA.

Dissolved in a little boiling dilute HCl, added pure NaHO in excess, boiled and filtered :—

<table>
<tr><td rowspan="2"><i>Filtrate</i>: added AmCl in excess, no pp. :—
<i>Abs. of Al.</i></td><td colspan="2"><i>Pp.</i>: dried and fused on platinum foil with Na_2CO_3 and KNO_3, boiled the colourless (<i>abs. of Cr</i>) mass when cold with water; decanted from the undissolved residue :—</td></tr>
<tr><td><i>Residue</i> in the dish dissolved by boiling with a little HCl, added several drops of KCyS: a blood-red coloration:
<i>Pres. of Fe.</i></td><td><i>Sol$^{n.}$</i> being colourless proved abs. of Cr.
Acidified with $H\bar{A}$, boiled, and added $Pb\bar{A}_2$, no yellow pp. :—
<i>Abs. of Cr.</i></td></tr>
</table>

y dissolve, the sol$^{n.}$ was decanted, and a little dilute HCl poured
melt. Added a little strong HCl, boiled as long as any smell of
ol$^{n.}$ and mixed it with the water sol$^{n.}$ which had been proved

<table>
<tr><td colspan="3">as, filtered :—</td></tr>
<tr><td colspan="3">s pink (<i>prob. pres. of Co</i>).
some strong HNO_3 and boiled to dryness (the colour of the sol$^{n.}$ changed to light brown
d the residue with HCl, it dissolved completely (<i>Abs. of H_2SiO_3</i>). Added a small portion
d; no pp.: <i>Absence of $(PO_4)'''$</i>. To the rem$^{r.}$ added AmCl then excess of AmHO, and</td></tr>
<tr><td colspan="3">te was again pink (<i>presence of Co</i>): added much Am_2S and boiled, filtered :—</td></tr>
<tr><td rowspan="2">ck pp.
m$^{d.}$ by Table III.B.</td><td colspan="2"><i>Filtrate</i> was yellow (<i>absence of Ni</i>): added Am_2CO_3, filtered :—</td></tr>
<tr><td><i>A white pp.</i>
Exam$^{d.}$ by Table IV.</td><td><i>Filtrate.</i>
Exam$^{d.}$ by Table V.</td></tr>
</table>

EXAM$^{N.}$ OF PP. IN GROUP III.B.

Rinsed the pp. off the filter with some cold dilute HCl, stirred well, filtered :—

<table>
<tr><td rowspan="2"><i>Pp.</i> was black and had already been proved to contain no Ni; fused a portion into a clear borax bead: bead blue in both flames :—
<i>Pres. of Co.</i></td><td colspan="2"><i>Filtrate:</i> boiled until it no longer smelt of H_2S, added a crystal of $KClO_3$, boiled until the smell of Cl ceased, cooled and added pure NaHO in excess :—</td></tr>
<tr><td>No pp.
<i>Abs. of Mn</i></td><td>Passed H_2S into the sol$^{n.}$ no pp :—
<i>Abs. of Zn.</i></td></tr>
</table>

EXAM$^{N.}$ OF PP. IN GROUP IV.

1. *Exam$^{n.}$ by Flame col$^{n.}$*—Dissolved a small quantity of the pp. in a few drops of HCl upon a watch glass, dipped a loop of Pt-wire into the sol$^{n.}$ and held it in the Bunsen flame: a crimson red colour was imparted to the flame, which appeared intense red through the indigo-prism: *Pres. of Sr.* This coloration was followed by a yellowish green very persistent col$^{n.}$: *Pres. of Ba.* Confirmed by spectroscope.

2. *Exam$^{n.}$ in the Wet way.* Dissolved the rest of the pp. in as little boiling $\overline{HA}$ as possible; to a small part of the solution, perfectly cold, added $CaSO_4$ sol$^{n.}$ an immediate pp. formed: *Pres. of Ba.*

To the remainder of the $H\bar{A}$ sol$^{n.}$, proved to be acid to litmus, added K_2CrO_4 until the liquid appeared yellow, warmed and poured through a double filter:—

<table>
<tr><td rowspan="2"><i>Pp.</i>, which contained all the Ba present, was rejected.</td><td colspan="2">To the clear <i>filtrate</i>, which was orange red in colour, added AmHO until the colour changed to light yellow, then added Am_2CO_3 in excess and filtered:—</td></tr>
<tr><td><i>Filtrate</i> was rejected.</td><td><i>Pp.:</i> dissolved in as little boiling $H\bar{A}$ as possible; added to a small portion of the sol$^{n.}$ $CaSO_4$ sol$^{n.}$ and boiled, a pp. formed, showing <i>pres. of Sr</i>.
To the remainder of the $H\bar{A}$ solution added H_2SO_4, boiled, filtered, and to the filtrate, which gave no pp. after being boiled with more H_2SO_4, added AmHO in excess and $Am_2C_2O_4$, no pp.:—
<i>Abs. of Ca.</i></td></tr>
</table>

Exam$^{N.}$ of Filtrate from General Table for Group V.

Evaporated the filtrate to dryness in a porcelain dish, scraped out the residue upon platinum foil, and ignited strongly until white fumes ceased to appear: dissolved the residue off the foil by boiling with water to which several drops of HCl had been added, divided the sol$^{n.}$ into two unequal parts:—

To the larger portion added several drops of H_2SO_4 and boiled, then AmHO in excess and several drops of $Am_2C_2O_4$; on heating, a slight pp. formed; filtered, and added to the clear filtrate Na_2HPO_4, a white crystalline pp.:— *Pres. of Mg.*	*Into the smaller portion* dipped a loop of platinum wire, and held it in the Bunsen flame, an intense yellow col$^{n.}$:— *Pres. of Na,* appearing crimson through the indigo-prism:— *Pres. of K.* Confirmed the pres. of K by stirring a fresh portion of the sol$^{n.}$ with $PtCl_4$ on a watch glass, a yellow pp. formed.

Metals found:—Cu, Fe, Co, Ba, Sr, Mg, Na, K, NH_4.

EXAM^N. FOR ACID-RADICLES.

The following acid-radicles have already been detected: $(CO_3)''$, $(NO_3)'$, $(ClO_3)'$.

By reference to the Table of Solubilities (440), under the columns corresponding to the above metals, the only salt insoluble in acids is found to be $BaSO_4$; since Ba is present, and the substance is entirely soluble in acids, (SO_4) *is absent.*

The following acid-radicles have also been proved to be absent in the general examination for metals :—

$(CrO_4)''$, by the HCl solution not becoming green on passing H_2S.

$(AsO_3)'''$ and $(AsO_4)'''$, by no pp. forming in Group II.B.

$(SiO_3)''$ and $(SiF_6)''$, by no residue insol. in HCl remaining on evaporating to dryness with HCl for Group III.A.

$(PO_4)'''$, by testing with $AmHMoO_4$ in the HCl sol^n. for Group III.A.

The organic acid-radicles $\bar{A}$ and $\bar{T}$ are absent, since there was no smell of burning on heating the substance.

The only commonly occurring acid-radicles remaining to be specially tested for are therefore Cl, Br, I, $(C_2O_4)''$, and $(BO_3)'''$; and of these Br and I are probably absent, since no violet fumes of I or brown fumes of Br were evolved with strong H_2SO_4.

Boiled a portion of the substance with pure Na_2CO_3 sol^n. and filtered; acidified portions of the filtrate with—

HNO_3	$H\bar{A}$
Added $AgNO_3$, a perfectly white pp., easily sol. in AmHO :— *Pres. of Cl.*	*Added* $CaSO_4$, no pp. :— *Abs. of* $(C_2O_4)''$.

Moistened a piece of turmeric paper with the HCl solⁿ· of the substance and dried it at 100°, no reddish brown stain was produced:—*Abs. of* $(BO_3)'''$.

Acid-radicles found:—CO_3, NO_3, ClO_3, Cl.

Found { *Metallic-radicles:* Cu, Fe, Co, Ba, Sr, Mg, Na, K, NH_4.
Acid-radicles: CO_3, NO_3, ClO_3, Cl.

REACTIONS FOR SOME OF THE RARER ELEMENTS.

The most important reactions for many of the rarer elements are given here. The arrangement is that adopted in the Fourth Section, those elements being placed together which are precipitated in the same analytical group: the groups, however, are arranged in the order in which they occur in the General Table. A scheme for the detection of these elements follows, showing in which group they are precipitated in the General Table, and by what reactions they will be most readily found.

GROUP I.—SILVER GROUP.

This group includes Tl and W: the former is partially precipitated as chloride by HCl, the latter completely as tungstic acid. Thallium is only partially precipitated in Group I. since its chloride is not quite insoluble in water: it therefore belongs also to Group III.B., being completely precipitated by Am_2S.

THALLIUM (Tl).—Use Tl_2SO_4 solution.

489. Tl occurs in small quantity in many natural sulphides, often also in the ashes of plants and in mineral waters. Tl yields both thall*ic* and thall*ious* salts, but the former are very unstable, changing even when their solutions are heated into thall*ious* salts.

1. *HCl:* a white precipitate, which rapidly settles, does not blacken in the light, and is soluble in aqua regia. It is soluble in a large quantity of water, and therefore does not form in dilute solutions.

2. *KI:* a yellow precipitate; almost insoluble in water, more soluble in KI solution. In a solution containing Fe, any ferric salt present must first be reduced by H_2SO_3 before adding KI.

3. *$PtCl_4$:* orange red precipitate, slightly soluble in water.

4. *Am_2S:* black precipitate, which is easily coagulated by heat, is insoluble in AmHO, in alkaline sulphides, and in KCy; it is readily

oxidised by the air to Tl_2SO_4, and is easily soluble in mineral acids. Tl is completely precipitated by H_2S from a solution in which $H\bar{A}$ is the only free acid present, but free mineral acids prevent the precipitation entirely.

5. *Flame coloration.*—Thallium compounds impart to the Bunsen flame an intense *green* colour, which, however, rapidly disappears. The spectrum (p. 66) is very characteristic, consisting of one bright emerald green line. Thallium may be easily detected by the spectroscope in solution, or better in any of its precipitates mentioned above.

TUNGSTATES.—Use Na_2WO_4 solution.

490. W usually occurs in the form of a tungstate. The insoluble tungstates yield soluble alkaline tungstates on being fused with alkaline carbonates. From the solution of an alkaline tungstate HCl precipitates the tungstic acid entirely.

1. *HCl, HNO_3 or H_2SO_4:* white precipitate (H_2WO_4), becoming yellow on boiling: insoluble in excess of acid, but soluble in AmHO. A piece of Zn dropped into the acid liquid containing the precipitate yields a deep blue colour.

2. *$SnCl_2$* in neutral solution, made by dissolving $SnCl_2$ crystals in water and filtering: *yellow* precipitate, becoming *blue* on addition of HCl and heating.

3. *Am_2S* yields no precipitate in the solution of an alkaline tungstate; but if, after adding Am_2S, the liquid is made acid with HCl, brown WS_3 is precipitated.

4. *Microcosmic bead:* outer flame, *colourless or yellow:* inner flame *blue,* if a little $FeSO_4$ is fused into the bead the colour changes to *blood red.* These colours are best seen when the bead is perfectly cold.

GROUP II.A.—COPPER GROUP.

In this group are included Pd (Os, Rh, Ru): they are precipitated as sulphides by H_2S from acid solutions, and the sulphides are insoluble in Am_2S and in caustic alkali solution.

PALLADIUM (Pd).—Use $PdCl_2$ solution.

491. Pd occurs as a metal in native platinum, also in gold and silver. Palladium solutions are reddish brown, or yellow if dilute:

addition of water precipitates a basic salt, unless sufficient free acid is present to prevent it.

1. *H_2S:* black precipitate, in neutral, alkaline, and acid solutions, insoluble in Am_2S, but soluble in boiling HCl or in aqua regia.

2. *AmHO:* flesh coloured precipitate ($PdCl_2.2NH_3$); soluble in excess of AmHO to a colourless liquid, from which HCl precipitates yellow crystalline paladammonium chloride ($N_2H_6Pd''Cl_2$).

3. *$HgCy_2$:* yellowish white gelatinous precipitate ($PdCy_2$), slightly soluble in HCl, easily soluble in AmHO. This is a very characteristic reaction.

4. *KI:* black precipitate (PdI_2): very characteristic of Pd.

GROUP II.B.—ARSENIC GROUP.

In this group are included Mo, Se, Te (Ir).

MOLYBDATES (Mo).—Use Am_2MoO_4 solution.

492. Mo occurs as molybdate; also as sulphide, which may be readily converted into MoO_3 by ignition in the air or by heating it with HNO_3. Unignited MoO_3 dissolves in acids; ignited MoO_3 is insoluble in acids, but easily soluble in alkalis.

1. *HCl, HNO_3, or H_2SO_4,* if added in small quantity to an aqueous solution of a molybdate, yields a precipitate which is readily soluble in excess of the acid.

2. *H_2S,* added in *very small* quantity to the acidified solution, gives a *blue* liquid; if it is added in larger quantities a *brown* precipitate (MoS_3) forms; the precipitation becomes complete only when the solution is heated and H_2S is passed for some time: the precipitate is soluble in solutions of alkaline sulphides and hydrates, from which it s reprecipitated when acid is added in excess.

3. *Zn or $SnCl_2$,* if added to the solution of a molybdate in HCl, colours it *brown, green, or blue,* according to its state of concentration.

4. *KCyS,* if added to a solution acidified with HCl, gives no coloration; but on dropping in a piece of Zn a beautiful *crimson* colour is produced, which, when the liquid is shaken with ether, is taken up by the ether.

5. *Na_2HPO_4,* if added in very small quantity to the solution of a molybdate acidified with HNO_3, gives, on gently warming the

liquid, a yellow precipitate readily soluble in excess of alkali hydrate solution.

6. *Borax bead:* outer flame, *yellow;* inner, *dark brown.*

7. *Microcosmic bead:* outer and inner flames, *green.*

SELENIUM (Se).

493. Se occurs in metallic selenides of Pb, Fe, Cu, Ag.

Selenides.—Use FeSe.

1. A selenide when heated in an open tube evolves a smell of decaying horse radish, and produces a grey or red sublimate of Se: the smell is very characteristic.

Selenites.—Use K_2SeO_3 (see 6, below).

2. *H_2S* gives in acid solutions, if cold a *yellow,* if hot a *reddish yellow* precipitate: soluble in Am_2S.

3. *$BaCl_2$* gives in neutral solutions a white precipitate: soluble in HCl and in HNO_3.

4. *$SnCl_2$ or H_2SO_3* gives in the presence of free HCl a red, or in warm solutions a grey precipitate of Se.

5. *Cu* in a hot HCl solution becomes covered with a black film: the liquid on standing with the Cu for some time becomes coloured red with Se.

Selenates.—Use K_2SeO_4.

6. *HCl* produces no change in the cold: but on boiling, Cl is given off and the selenate is reduced to selenite, to which the above tests may be applied.

7. *$BaCl_2$:* a white precipitate ($BaSeO_4$); insoluble in cold HCl, dissolved by boiling with HCl with evolution of Cl and reduction to $BaSeO_3$.

Selenium in any form of combination.

8. Heated on charcoal in the inner blowpipe flame a smell of rotten horse radish is perceived.

9. Fused on charcoal with Na_2CO_3 in the inner blowpipe flame a fused mass is obtained, which when moistened on Ag yields a black stain, and on addition of HCl evolves H_2Se, a badly smelling gas.

TELLURIUM (Te).

494. Te commonly occurs united with the metals Au, Ag, Bi, Cu, Pb.

Tellurides.—Use PbTe.

1. A telluride when heated in an open glass tube gives white fumes

and a sublimate which differs from that given by Sb, by being fusible before the blowpipe.

Tellurites.—Use K_2TeO_3 (see 5, below).

2. *H_2O:* on dilution with water tellurous acid is precipitated from the acid solution.

3. *H_2S:* in acid solutions a brown precipitate (TeS_2); easily soluble in Am_2S.

4. *H_2SO_3, $SnCl_2$, or Zn* precipitates black Te.

Tellurates.—Use $KeTeO_4$.

5. HCl produces no change in the cold solution of a tellurate; but on boiling, Cl is evolved and the tellurate is reduced to tellurite; the solution then gives the above reactions.

Tellurium in any form of combination.

6. A tellurium compound if fused with Na_2CO_3 on charcoal in the inner blowpipe flame, gives sodium telluride; a solution of which gives a black stain on Ag, and when it is acidified deposits black Te and evolves H_2Te.

GROUP III.—IRON AND ZINC GROUPS.

In this Group are included U, In, Ti, Be, Tl, [V], (Zr, Ce, Ta, Nb, La, Di, Y, E, Th).

Tl is often partially precipitated as chloride in Group I., and its reactions are given under that group. V is not precipitated by Am_2S unless acid is added in excess after Am_2S.

URANIUM (U).—Use $(UO_2)''(NO_3)_2$.

495. U occurs in nature principally as *pitchblende,* an oxide, also as *uranite,* a hydrated uranium calcium phosphate and *chalcolite* hydrated uranium copper phosphate.

1. *AmHO, KHO, or NaHO:* yellow precipitate, insoluble in excess.

2. *Am_2S* gives in neutral solutions a *dingy yellow* or *brown* precipitate of uranium oxysulphide, which differs from ZnS, MnS and FeS, by being soluble in Am_2CO_3. The precipitate settles slowly unless AmCl is added; it is soluble in acids, even in $H\bar{A}$: on being heated with Am_2S in excess it is changed into uranous oxide and sulphur.

3. *H_2S* produces no precipitate in acid solutions.

4. *Am_2CO_3, $KHCO_3$, or $NaHCO_3$:* yellow precipitate easily soluble in excess; from this solution the uranium is reprecipitated by addition of NaHO or KHO, or by boiling, differing thus from Fe.

5. K_4FeCy_6 gives in acid solutions a reddish brown precipitate, which is distinguished from the similar one produced in a copper solution by dissolving in excess of AmHO to a *yellow* fluid.

6. $BaCO_3$ causes complete precipitation even in the cold.

7. *Zn* changes the *yellow* colour of acid solutions to *green.*

8. *Borax and microcosmic beads:* outer flame, *yellow;* inner flame, *green.*

INDIUM (In).—Use $In_2(SO_4)_3$.

496. Occurs in zinc blende and in wolfram.

1. *Alkali hydrates* precipitate a hydrate resembling $Al_2H_6O_6$, insoluble in excess. Indium solutions are also precipitated by alkaline carbonates, by Na_2HPO_4, by boiling their neutral solutions with excess of $Na\bar{A}$, by $BaCO_3$, and by alkaline oxalates.

2. H_2S gives no precipitate in strong and acidified solutions, in dilute and feebly acid solutions a little sulphide separates, in a solution containing no free acid but $H\bar{A}$ the metal is entirely precipitated as *yellow* sulphide: the precipitate is insoluble in cold, but soluble in boiling Am_2S; from the boiling solution *white* sulphide separates on cooling.

3. Am_2S, added after $H_2\bar{T}$ and excess of AmHO, gives a white precipitate, becoming yellow on treatment with $H\bar{A}$.

4. *Flame coloration:* bluish violet. The spectrum shows a very characteristic blue line, which is brilliant, but very rapidly disappears when the chloride is employed: see page 66.

BERYLLIUM (Be).—Use $Be_2(SO_4)_3$.

497. Be occurs as silicate in phenakite, and as silicate with Al-silicate in beryl and emerald.

1. Am_2S, *AmHO, KHO, or NaHO:* the flocculent hydrate is precipitated, resembling $Al_2H_6O_6$ in appearance, and in being soluble in KHO: but it differs from $Al_2H_6O_6$ in being precipitated from its solution in KHO by dilution and long boiling, and by being soluble when freshly precipitated if it is long boiled with AmCl solution.

2. *Alkaline carbonates* precipitate a carbonate, which is soluble in excess, especially in Am_2CO_3; from these solutions the carbonate is reprecipitated on diluting and boiling for some time, and with especial ease from the Am_2CO_3 solution, differing thus from Al.

3. $BaCO_3$ precipitates Be-solutions completely.

4. $H_2C_2O_4$ and alkaline oxalates produce no precipitate.

5. Moistened with $Co(NO_3)_2$ solution and heated on charcoal in the *outer* blowpipe flame, a *grey* mass is obtained, difference from Al.

TITANIUM (Ti).—Use TiO_2.

498. Ti occurs as TiO_2, with traces of Fe, Mn, Cr, in rutile, anatase, and brookite: also as TiO_2 in combination with Fe in titaniferous iron ore.

1. Ignited TiO_2 is insoluble in water and in most acids: it is easily soluble in HF, less readily in boiling strong H_2SO_4: it is also rendered soluble in cold water by fusion with $KHSO_4$. TiO_2 differs from SiO_2 in not being volatilised when heated in a platinum dish with HF and strong H_2SO_4.

2. *By dilution and long boiling*, white flocculent hydrated TiO_2 is precipitated from solutions in H_2SO_4 or HCl, and from the aqueous solution of the fusion with $KHSO_4$: the precipitate is metatitanic acid: it cannot be filtered off unless an acid or AmCl is added.

3. *AmHO, KHO, NaHO, Am_2S, or $BaCO_3$*, white flocculent precipitate, insoluble in excess: if precipitated and washed in the cold it dissolves in HCl and in dilute H_2SO_4.

4. *Zn or Sn* gives in acid solutions a *blue*, or if dilute a *rose red* coloration.

5. *$Na_2S_2O_3$*, on boiling, precipitates Ti-solution entirely, difference from Fe.

6. *Microcosmic bead:* in the outer flame *yellow* while hot, *colourless* when cold: in the inner flame *yellow* while hot, *violet* when cold.

The production of these colours is much aided by adding a fragment of Sn. Addition of a small quantity of $FeSO_4$ gives in the inner flame a *blood red* bead.

VANADIUM (V).—Use $NaVO_3$.

499. V occurs in vanadinite [$3Pb_3(PO_4)_2.PbCl_2$], and in certain Fe- and Cu-ores. Vanadium is known in several stages of oxidation; it will usually occur in analysis as vanadic acid or a vanadate, which in acid solution is of a yellow or reddish colour.

1. *H_2S, H_2SO_3, or $H_2C_2O_4$* reduces acid solutions of a vanadate, and causes the colour of the solution to change to *blue:* with H_2S a deposit of sulphur also forms.

2. *Am_2S*, if added in excess, gives a brown liquid, in which an excess of acid causes a *brown* precipitate of V_2S_5; excess of Am_2S dissolves this to a reddish brown liquid.

3. *Zn* gives in a very dilute solution acidified with H_2SO_4 when gently warmed a *blue* colour, which changes into *green* and then *lavender blue.*

4. *Solid AmCl*, added until the solution is saturated, precipitates Am_3VO_4, which is insoluble in saturated AmCl solution. This is a very characteristic reaction.

5. H_2O_2 when shaken up with an acid solution imparts a red colour which is not removed by shaking with ether. This is a very delicate test.

6. *Borax bead:* in outer flame *colourless*, or *yellow* if much V is present: in inner flame, *green* hot and cold, or if much V is present *brown* hot and *green* cold.

GROUP V.—POTASSIUM GROUP.

In this group are included Li, Cs, Rb.

Lithium (Li).—Use LiCl.

500. Li occurs frequently in mineral waters and in the ashes of plants, also in small quantities in the minerals lepidolite, triphane, and petallite.

It is allied to the metals of the Barium Group by the difficult solubility of its carbonate and phosphate; and it differs from K and NH_4 by not being precipitated by $PtCl_4$ or by $H_2\overline{T}$: from Na it is readily distinguished by the flame coloration and spectroscope.

LiCl is separated from KCl and NaCl by its insolubility in a mixture of absolute alcohol and ether.

1. Na_2HPO_4 added to the solution, which must not be too dilute and must be strongly alkaline with NaHO, gives, on boiling, a white crystalline precipitate (Li_3PO_4) which quickly settles. Traces of Li may be precipitated by adding Na_2HPO_4, then NaHO until the liquid remains alkaline, evaporating to dryness and washing the residue with dilute AmHO.

This precipitate differs from the phosphates of Ba, Sr, Ca, and Mg by fusing in the blowpipe flame and being absorbed by the charcoal support, also by its diluted solution in HCl giving no precipitate on addition of AmHO in excess in the cold, but a white crystalline precipitate on boiling.

2. *Flame coloration:* this is *carmine red;* the spectrum (page 66) consists of an intense carmine red line (α). This coloration is concealed by that of Na, which, however, does not interfere with the spectrum.

The Na coloration is also removed if the flame is viewed through the indigo prism, whereas that of Li can pass through the thinner parts of the prism; it differs from the K coloration, however, in being unable to penetrate the thick layers of indigo solution or in being much weakened by its passage.

As silicate, Li only gives the flame coloration after fusion with $CaSO_4$; the phosphate requires to be first moistened with HCl.

RUBIDIUM (Rb), CÆSIUM (Cs).—Use RbCl and CsCl.

501. These metals occur in small quantities in some mineral waters and in a few minerals.

1. Their compounds resemble those of K by being precipitated by $PtCl_4$ and by $H_2\overline{T}$, and by giving a similar flame coloration. Characteristic differences are the much greater insolubility in water of the platino-chlorides, which enables the K_2PtCl_6 to be dissolved away from Rb_2PtCl_6 and Cs_2PtCl_6 by boiling the precipitate with successive small quantities of water: the alums also show a similar difference in solubility in cold water.

2. Cs and Rb are readily distinguished from one another and from other elements by their spectra, see page 66. For Cs the blue lines (α, β) are especially distinct and characteristic: in the Rb spectrum the indigo blue lines (α, β) are very distinct, but the red lines (γ, δ) are most characteristic. For the spectroscopic test the chlorides are most suitable.

502. GENERAL GROUP TABLE FOR

Group I.—Reagent HCl.	*Group II.*—Reagent H_2S.
The group pp. may contain in addition to $AgCl$, Hg_2Cl_2, $PbCl_2$:— *TlCl* (*white*). H_2WO_4 („). *TlCl* will usually be readily detected by a spectroscopic examination of the group pp. It may be removed from the group pp. by boiling with a little water, and pp$^{d.}$ from the cold filtrate by KI (Pb is also thus pp$^{d.}$): the pp. is yellow, and gives the thallium spectrum. H_2WO_4: a white pp. becoming yellow on boiling. Its presence is confirmed by dropping a piece of Zn into a portion of the acid liquid and pp., when a *deep blue* colour will be obtained. Also by fusion of the pp. in a microcosmic bead, which will be *colourless* or *yellow* in the outer flame, *blue* (or with $FeSO_4$, *blood-red*) in the inner flame.	*The group pp. may contain as sulphides, in addition to Hg, Pb, Bi, Cu, Cd :—* *Pd* (*black-brown*) *Tl* (*black; pp$^{d.}$ with As_2S_3*) [*Os*, *Rh*, *Ru.**] } Insoluble in Am_2S. *And in addition to SnS, SnS_2, Sb_2S_3, As_2S_3, Au_2S_3, PtS_2 :—* [*Ir*-sulphide*] *Mo-* „ (*brown* *Se-* „ (*red-yellow*) *Te-* „ (*black*) } Soluble in Am_2S. The *yellow* colour of a solution containing V is changed to *blue* by H_2S. *Tl* is readily found by the spectroscope in the group pp. *Pd* will remain in solution in excess of AmHO with Cd and Cu in Table II.A: from this solution it is pp$^{d.}$ by adding HCl: its presence may be further confirmed by the $HgCy_2$ or KI tests (491). The examination of the sulphides in Group II.B when the above elements may be present is best commenced by fusion with Na_2CO_3 and $NaNO_3$. From the fused mass, water dissolves sodium-arsenate, -molybdate, -selenate, and -tellurate, leaving SnO_2, sodium-antimonate, Au, Pt, and Ir undissolved; the elements may then be detected by special tests. For the separation and detection of the platinum metals a larger work must be consulted. * Only completely pp$^{t.}$ if the liquid has been warmed, and H_2S passed for a long time.

PRECIPITATION OF RARER ELEMENTS.

Group III.—Reagents AmCl, AmHO, and Am_2S.	Precipitated from the filtrate from Group III. by excess of HCl.	*Group V.*
The group pp. may contain as sulphides, in addition to Fe, Zn, Mn, Ni, Co:— *U (black brown.)* *In* (,, ,,) *Tl* (,, ,,) *And as hydrates in addition to Al, Cr (and phosphates, &c. :)—* *Ti (white, flocculent.)* *Be (colourless, gelatinous).* [*Zr* ,, ,,] [*Ta, Nb, Ce, La, Di, Y, E, Th.*] *Tl* and *In* will usually be readily detected by a spectroscopic examination of the pp. *Tl* may be first separated and detected by dissolving some of the group pp. in boiling dilute HCl, and reducing any Fe present in the solution by H_2SO_3; then precipitating Tl by addition of KI, and testing the pp. by the spectroscope. A more complete scheme for the analysis of this group pp. will be found in par. (503).	*As sulphides*:— *V (black).* *W* (trace, *brown*). *Ni* (trace, *black*). This pp. is dried and then fused with a mixture of Na_2CO_3 and KNO_3: on boiling the mass with water, NiO remains undissolved; it is filtered off, and its presence confirmed by fusion in a borax bead. The aqueous solution may contain alkaline vanadate and tungstate. V is separated by saturating the liquid with AmCl, and its presence confirmed in the pp. by the borax bead, and by dissolving the pp. in HCl and employing the reactions with H_2O_2 and with Zn (499). The filtrate, after concentration if necessary, is acidified with HCl, and the presence of W confirmed by the Zn reaction and the microcosmic bead (490).	The filtrate after the separation of all the group precipitates may contain besides Mg, K, and Na :— *Li, Cs, Rb.* These metals are readily detected by their very characteristic spectra (p. 66). LiCl may also be detected by its behaviour with Na_2HPO_4. Before proceeding to the spectroscopic examination, it is well to convert them into chlorides if they are not already in that form. If the chlorides are evaporated to dryness and extracted with absolute ether and alcohol, to which a few drops of HCl have been added, LiCl, RbCl, CsCl dissolve, whilst the greater part of the KCl and NaCl remains undissolved. The solution is evaporated to dryness, dissolved in a little HCl, and Rb and Cs are pp$^{d.}$ by $PtCl_4$. this pp. is boiled with small quantities of water until it no longer gives the K spectrum, the spectra of Rb and Cs will then be seen, if present. Li is found in the filtrate after adding $PtCl_4$.

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503.—Table for Analysis of Group III. Precipitate,

The precipitate may contain Fe, U, In, Tl, Al, Cr, Zn,

<table>
<tr><td colspan="4">After a preliminary spectroscopic examination has been made of a small portion of the the General Table (502), the group pp. is dried and fused in platinum with $KHSO_4$ for long time with cold water:—</td></tr>
<tr><td rowspan="6"><i>Residue:</i> may contain Ta_2O_5, Nb_2O_5, also SiO_2, and traces of Fe and Cr which have escaped solution. By fusion with $KClO_3$ and NaHO, Cr and Si are rendered soluble in dilute NaHO; from the residue, insoluble in NaHO, Ta_2O_5 and Nb_2O_5 may be removed by washing with dilute Na_2CO_3 solution.</td><td colspan="3"><i>Solution:</i> Reduce Fe by adding H_2SO_3 in excess, dilute consider- covering the vessel, and by passing a stream of CO_2</td></tr>
<tr><td rowspan="5"><i>Pp.</i> may consist of <i>TiO_2</i>, possibly also traces of Zr.
Confirm the presence of TiO_2 by the microcosmic bead (498).</td><td colspan="2"><i>Filtrate:</i> add a few drops of strong HNO_3, and precipitate once more by adding excess</td></tr>
<tr><td colspan="2"><i>Pp.</i> may contain Fe, In, U, Cr, Al, Be, &c.</td></tr>
<tr><td colspan="2"><i>Pp.</i> may contain Fe, In, U, &c. Dissolve large excess of HCl, add $BaCO_3$, and let</td></tr>
<tr><td rowspan="2"><i>Pp.</i> may contain Fe, In, U, also traces of Al and Cr.
It is dissolved in HCl, and $NaHCO_3$ is added in excess, whereby U alone is obtained in solution.
In is found by the spectroscope:
Cr by fusion with Na_2CO_3 and $KClO_3$.</td><td><i>Filtrate</i> may contain Th. Ba is pp^d by H_2SO_4, the liquid exactly neutralised of K_2SO_4 are added, allowed to stand for pp. is washed with</td></tr>
<tr><td><i>Pp.</i> may contain Zr, Th, Ce.
Th and Ce are dissolved away by boiling with dilute HCl, and are repp^d by AmHO.</td></tr>
</table>

WHEN THE RARER ELEMENTS MAY BE PRESENT.

Mn, Ni, Co, Ti, Be, [Zr, Ta, Nb, Ce, La, Di, Y, E, Th].

<table>
<tr><td colspan="3">pp. for Tl and In, and Tl has been further tested for, if necessary, by KI as directed in
some time, the cold mass is then powdered and allowed to stand, with shaking, for a</td></tr>
<tr><td colspan="3">ably and boil for a long time, preserving the liquid from oxidation by the air by
unless it smells constantly of SO_2 :—</td></tr>
<tr><td colspan="3">concentrate by evaporation, and add AmHO in excess : filter, dissolve the pp. in HCl,
of AmHO :—</td></tr>
<tr><td colspan="2">Dissolve it in HCl, and add excess of cold strong KHO solution :—</td><td rowspan="4"><i>Filtrate</i> may contain :—
Zn, Mn, Ni, Co.</td></tr>
<tr><td>in HCl, boil off any
stand :—</td><td rowspan="3"><i>Filtrate</i> may contain Al, Cr, Be : dilute and boil for some time, filter. Al remains in solution, and may be $pp^{d.}$ by adding HCl just in excess, then AmHO in excess.
Cr and Be are separated by fusion with Na_2CO_3 and $KClO_3$, dissolving in water and $pp^{g.}$ Be by adding HNO_3 in excess, then AmHO in excess.</td></tr>
<tr><td>Zr, Ce, La, Di, V, E,
adding excess of
is concentrated and
with KHO. Crystals
and the liquid is
some hours. The
K_2SO_4 solution.</td></tr>
<tr><td><i>Sol</i>^{n}. may contain Y, E, also Be.
The solution is $pp^{d.}$ by AmHO, and Be dissolved away from it by $H_2C_2O_4$.</td></tr>
</table>

REACTIONS FOR CERTAIN ORGANIC SUBSTANCES.

I. ORGANIC ACID-RADICLES.

The most important reactions are given here for the more commonly occurring organic acid-radicles.

FORMATES.—Use $NaHCO_3$.

504. The acid and the salts much resemble acetic acid and acetates.

1. Fe_2Cl_6 imparts a deep red colour to the solution of a formate; the coloration is destroyed by the addition of HCl: on boiling the red liquid basic ferric formate is precipitated, and the liquid becomes colourless if excess of formate was present, since all the Fe is precipitated. This reaction is precisely similar to that for an acetate (328).

2. *$AgNO_3$, or $Hg_2(NO_3)_2$* solution when heated with solution of a formate, in the absence of free acid and alkali, yields precipitates of black Ag and Hg respectively.

Solution of $HgCl_2$, when heated to about 70° with the solution of a formate, yields a white precipitate (Hg_2Cl_2) if HCl and alkaline chlorides are absent.

3. *Strong H_2SO_4* when heated with a formate evolves CO gas, which burns with a blue flame. No charring or blackening occurs, and no evolution of CO_2 as in the case of an oxalate (317):

If a formate is distilled with dilute H_2SO_4, a distillate emitting the pungent smell of formic acid is obtained.

4. When a solid formate is strongly heated it fuses and chars, and evolves a mixture of CO and CO_2.

CITRATES.—Use $Na_3C_6H_5O_7.2H_2O$.

505. Citrates much resemble tartrates and oxalates in their reactions. Citrates differ, however, by emitting pungent acid fumes when they are carbonised by heat: and by darkening when they are heated with strong H_2SO_4, but only after the mixture has been heated for some time and inflammable gases have escaped.

Further, calcium citrate is only precipitated *after long standing or on boiling the liquid* when large excess of lime water or AmHO, AmCl and $CaCl_2$ have been added: the precipitate is insoluble in KHO.

Citrates are not precipitated by solution of $K\bar{A}$.

SUCCINATES, BENZOATES.—Use $Am_2C_4H_4O_4$ and $AmC_7H_5O_2$.

506. Succinic acid and succinates frequently smell of amber, while benzoic acid and benzoates commonly emit an aromatic smell of gum benzoin.

Succinates and benzoates closely resemble one another in their analytical reactions; the following differences are therefore important.

Succinic acid is readily soluble in water: benzoic acid is only slightly soluble in cold water, and is therefore partially precipitated from its solution in alcohol on the addition of water, or when strong HCl or any other strong acid is added to its aqueous solution.

Both acids when heated emit fumes which are very irritating to the throat, and which cause coughing when inhaled.

The addition of $BaCl_2$ solution and much alcohol precipitates succinates but not benzoates. A similar difference is shown with $CaCl_2$.

Fe_2Cl_6 gives a *reddish brown* precipitate with a succinate, and a *pale brown or buff* precipitate with a benzoate.

TANNATES. GALLATES.—Use the acids, $C_{14}H_{10}O_9$ and $C_7H_6O_5$.

507. These acids resemble one another in yielding *bluish black* precipitates with Fe_2Cl_6 solution. Tannic acid may be distinguished by yielding a flocculent precipitate with solution of gelatin or isinglass.

URATES.—Use the acid $C_5H_4N_4O_3$, or $AmC_5H_3N_4O_3$.

508. HNO_3 dissolves the acid with effervescence: if this solution is evaporated to dryness in a porcelain dish, and the residue is moistened with AmHO, bright purple *murexide* is formed.

SALICYLATES.—Use the acid, $C_7H_6O_3$.

509. Salicylic acid melts at 157°, and at a somewhat higher temperature decomposes into CO_2 and phenol or carbolic acid (C_6H_6O), the change being rapid and complete if the acid is mixed with excess of quicklime before it is heated: the phenol condenses as a white crystalline sublimate.

The solution of salicylic acid becomes *violet* on the addition of Fe_2Cl_6 solution.

MECONATES.—Use the acid, $C_7H_4O_7$.

510. Meconic acid is present in opium, and may be detected in extracts of that substance by the production of a *deep red* coloration on the addition of Fe_2Cl_6.

This coloration differs from that caused by an acetate or a formate by not disappearing when the liquid is boiled: and may be distinguished from ferric sulphocyanide by its colour not being destroyed when the liquid is dropped into solution of $HgCl_2$.

II. ALKALOIDS.

Most alkaloids resemble metals or rather NH_3 in their behaviour with acids, and may therefore be looked upon as basic.

CINCHONINE. QUININE.—Use cinchonine sulphate and $2C_{20}H_{24}N_2O_2.H_2SO_4.7H_2O$.

511. In dissolving quinine sulphate in water a few drops of dilute H_2SO_4 should be added. It is intensely bitter and gives a pale blue fluorescence: pure solution of cinchonine sulphate is not fluorescent.

1. Solution of alkaline hydroxide or carbonate precipitates hydrated quinine from fairly strong solutions of a quinine salt; this precipitate disappears when the liquid is shaken with ether.

The corresponding cinchonine precipitate is not thus dissolved by ether.

2. *Br- or Cl-water* does not colour a solution of quinine, but if AmHO is afterwards added an intense *emerald green* colour appears.

Cinchonine solution under these conditions yields a yellowish white precipitate.

3. *If Br- or Cl-water* is added to a quinine solution, then K_4FeCy_6 and one or two drops of KHO solution, a *deep red* tint is produced, which quickly changes to dirty brown: this colour is destroyed by $H\bar{A}$, but reappears when AmHO is cautiously added.

4. *Tannic acid* solution yields in quinine solution a curdy precipitate, which is readily dissolved by $H\bar{A}$.

MORPHINE.—Use $C_{17}H_{19}NO_3.HCl.3H_2O$.

512. This alkaloid is present in opium and opium extracts: it is prepared from opium.

1. *K_2CO_3, or $KHCO_3$*, or one of the corresponding sodium salts, precipitates from fairly strong solutions $\overline{Mo}.2H_2O$, which is insoluble in excess. KHO or AmHO yields the same precipitate, soluble in excess of the reagent.

2. *Strong HNO_3* produces a *yellowish red* colour, which does not change to *violet* on the addition of $SnCl_2$. Difference from brucine.

3. *Neutral Fe_2Cl_6* solution if added drop by drop to neutral morphine solution produces a *dark blue* colour: this disappears on the addition of an acid.

4. *From iodic acid* morphine and its salts separate iodine: this is

shown by the liquid becoming brown, but the iodine is more certainly and readily detected by adding starch solution, or by shaking the liquid with CS_2 (268): the brown coloration of the solution becomes more intense on the addition of AmHO.

The test is very delicate and characteristic if the solid substance is moistened with a solution of one part of iodic acid in fifteen of water, and solution of one part of starch in four hundred of water is added: on pouring very dilute AmHO upon the blue solution thus obtained a coloured ring is seen at the surface of contact of the solutions, which is *blue* below and *brown* above.

This reaction serves to distinguish morphine from other organic substances containing nitrogen.

5. H_2SO_4 gives no colour when added to a solution containing morphine, but on dropping a crystal of $K_2Cr_2O_7$ into the acid liquid contained in a white porcelain dish, and slowly moving the crystal about with a glass rod, an *intense green* colour appears.

6. If solid morphine or any of its compounds is dissolved by heating it with a few drops of strong H_2SO_4 for fifteen minutes upon the water bath, and a little H_2SO_4 containing HNO_3 (see below) is added to this solution a *violet red* colour appears: on dropping in a fragment of MnO_2 it becomes surrounded by an intense mahogany red coloration.

The acid mentioned above is prepared by mixing six drops of HNO_3 of 1·25 sp. gr. with 100 c.c. of water, adding ten drops of this mixture to 20 grams of pure strong H_2SO_4, and mixing twenty drops of this solution with two or three drops of water before adding them to the morphine solution.

STRYCHNINE.—Use $C_{21}H_{22}N_2O_2$.

513. In dissolving strychnine in water a drop of dilute H_2SO_4 should be added. The solutions are very poisonous: they possess an intensely bitter taste even when they are extremely dilute.

1. Strychnine dissolves in strong H_2SO_4 to a colourless liquid: on adding a fragment of $K_2Cr_2O_7$ to this solution in a porcelain dish and slowly moving the bichromate about by pushing it with a glass rod, a *bluish violet* coloration is produced, which gradually changes to red or yellow.

The presence of morphia, or of metallic chlorides or nitrates, interferes with this reaction: if any of these should be present, the strychnine may be precipitated by the addition of $K_6Fe_2Cy_{12}$ or of K_2CrO_4 solution, filtered off and slightly washed on the filter, and then stirred with the strong H_2SO_4.

2. *Cold strong* HNO_3 dissolves strychnine without becoming coloured, but the solution acquires a yellow tint when it is heated.

NARCOTINE.—Use $C_{22}H_{23}NO_7$, or its sulphate.

514. Narcotine is present in opium, but is not extracted by the processes usually employed.

1. *Strong H_2SO_4* gives a *bluish violet* coloration changing to orange, but in some specimens a yellow solution is produced at once.

When this liquid is *gradually warmed* it becomes first *orange red* and then *bluish violet*, or *purple* stripes proceed from the edge of the liquid surface: on cooling, the colour changes to *cherry red.* If the heating is pushed to the initial evaporation of the acid, an intense *reddish violet* colour is produced.

2. If narcotine is subjected to reaction (512. 6) for morphine, an intense red coloration is produced on the addition of the acid reagent, and this does not alter when MnO_2 is dropped in.

BRUCINE.—Use $C_{23}H_{26}N_2O_4$, or its sulphate.

515. The substance is most readily dissolved in water when a drop of H_2SO_4 is added.

1. *Strong HNO_3* yields an *intensely red* coloration, which gradually changes to yellowish red and yellow when the liquid is heated: when $SnCl_2$ is added to the hot solution it becomes intensely *violet.* Difference from morphine.

2. *With strong H_2SO_4* a *rose coloured* solution is obtained, which changes to *yellow.* On adding the acid reagent described in (512. 6), the liquid becomes red, but quickly changes to yellow.

III. CERTAIN OTHER ORGANIC SUBSTANCES.

ALCOHOL, or ETHYL ALCOHOL.—Use weak spirit.

516. Pure ethyl alcohol boils at 78°·4 under 760 mm. mercury pressure, and has a specific gravity of 0·7938 at 15°·5. It burns with a pale blue smokeless flame.

If the alcohol is very dilute it may be necessary to add Na_2CO_3 in excess to the liquid, and distil over about one-third of it: the alcohol is thus concentrated and is the more readily detected by tests 1 and 2.

1. If Na_2CO_3 solution is added to water containing a little alcohol and the mixture is digested for some time at a gentle heat, occasionally dropping in small pieces of iodine until the solution retains a brown

colour, *golden yellow crystals of iodoform* will separate after several hours: if very little alcohol is present, ten or twelve hours may be necessary for the formation of these crystals.

When examined under the microscope, the crystals are seen to be hexagonal tablets or six-pointed stars.

This reaction is yielded by other substances besides ethyl alcohol, and is therefore not distinctive.

2. Alcohol may be converted into $H\overline{A}$ by heating it for some time with $K_2Cr_2O_7$ and H_2SO_4. Aldehyde will be smelt first and then acetic acid. If the smell of the acid is not distinctly perceptible, the liquid may be distilled and the first portion of the distillate neutralised with Na_2CO_3: this solution is then evaporated to dryness and the solid residue is tested for an acetate (326–328).

Acetic acid may result from the oxidation of other substances besides alcohol.

STARCH.—Use potato or rice starch in powder, $(C_6H_{10}O_5)_n$.

517. Starch is insoluble in cold water, but passes into a transparent liquid when it is boiled with water; this liquid becomes gelatinous on cooling, if much starch is present.

1. Starch granules may be identified by their appearance when examined by a quarter inch object glass under the microscope: viewed as transparent objects, they appear round or oval with more or less concentric dark rings. Many kinds of starch granules show a cross when examined by polarised light.

These appearances are not visible in starch which has been exposed to heat.

2. If starch is boiled with water, the solution *when cold* assumes an intensely *bluish black* colour on the addition of a drop of solution of iodine: this colour disappears when the liquid is heated, but frequently reappears when it is cooled.

GRAPE SUGAR, OR GLUCOSE.—Use $(C_6H_{12}O_6)_n$.

518. Glucose is distinguished from other sugars by yielding only a yellow solution with strong H_2SO_4 in the presence of a little water if the mixture is kept cool: other sugars become black by charring.

Glucose when warmed with strong KHO solution becomes *brown*, while other sugars remain uncoloured.

1. If a few drops of $CuSO_4$ solution are mixed with glucose solution and excess of KHO is then added, no precipitate is formed, but when the liquid is boiled red Cu_2O is precipitated.

3. If clean freshly washed yeast is added to glucose solution and the liquid is kept at a temperature of about 25° for an hour or more in the

flask shown in fig. 44 (page 138), fermentation will take place with the production of ethyl alcohol and carbon dioxide gas.

The escaping gas may be detected by letting it bubble through lime water (229. 4), and ethyl alcohol may be found in the liquid which remains in the flask by the reactions given in paragraph (516).

UREA.—Use CH_4N_2O.

519. Urea resembles the alkaloids in directly combining with acids to produce salts.

1. *Strong* HNO_3, if added to solution of urea, causes the separation of the crystalline nitrate: under the microscope these crystals are seen to be delicate rhomboidal scales.

2. *KHO* when heated with urea solution slowly evolves NH_3, and yields K_2CO_3, which effervesces on the addition of an acid.

3. $Hg(NO_3)_2$ yields a white precipitate in urea solution.

ALBUMEN.—Use white of egg.

520. Solution of albumen is readily obtained by stirring or shaking the fresh unboiled white of egg with water and straining the solution through linen or allowing it to settle.

1. Albumen is readily coagulated and precipitated as white flocks from its solution in water either by boiling the solution, or by adding it to strong HNO_3, or solution of $HgCl_2$. Solution of $CuSO_4$, alum, or of other salts, precipitates albumen from its solution less completely than $HgCl_2$ does.

SECTION VII.

LABORATORY FITTINGS, APPARATUS, CHEMICALS AND REAGENTS.

In this Section such directions are given as have been found by experience to be of value in fitting and furnishing a laboratory and in maintaining it in working order.

LABORATORY FITTINGS.

530. The building, fitting, heating and ventilation of a chemical laboratory are matters of prime importance in securing the comfort, health and convenience of the laboratory student, and in recent years very considerable advances have been made in these matters. For detailed information on these subjects, reference should be made to papers by E. C. Robins, which are published in the *Transactions of the Institute of British Architects* for the years 1882-3 and 1883-4.

With regard to the building, it must suffice to state here that the main points requiring attention are that the internal air space, ventilation, and window lighting both from the top and sides, should be secured on as ample a scale as possible. The inner surface of the walls should either be of glazed brick, which can be easily cleaned when necessary, or the rough brick or plaster surface should be covered with lime wash or distemper, which can be frequently and cheaply renewed.

Around the walls, in the spaces between the windows, there should be large and small recesses, each of which is lined with white glazed tiles, has a glazed hood above and is closed with a sliding sash in front; it is provided at the top with an outlet, through which a strong draught is caused. These draught cupboards should be provided with several gas supplies for Bunsen burners, the taps being on the outside of the cupboard: they should also be furnished with a gas jet for lighting the interior when necessary. The bottom of the cupboard may be covered with sheet-lead, and provided with a small grid through which liquids escape into a drain pipe. Some at least of these cupboards should also be provided with a water supply pipe. A strip of india-rubber draught-excluder fixed along the top of the inner frame and lightly touching the glass, prevents the escape of fumes when the sash is partly opened.

One or more of the larger draught cupboards will be reserved for the sulphuretted hydrogen apparatus (534): it will be found well to provide such a cupboard with small doors through which the gas may be utilised, so as to prevent it from escaping in large quantity into the laboratory by opening the large sash. A frame containing pairs of little hinged glazed doors, each about six inches by eight, may be introduced beneath the sliding sash, and this enables a student to open as much of the cupboard as is absolutely necessary for introducing his solution: while the whole sash can be raised for cleansing or replenishing the sulphuretted hydrogen apparatus.

One of the draught recesses is kept open for the steam ovens, small boiler and condenser which is described in (535).

Two or more deep box sinks covered with sheet-lead should be provided for general use; these have large water taps fixed above them at some distance from the back of the sink. It will be found convenient if each sink is supplied with three taps, two of which deliver low pressure water, hot and cold, and a third is connected with a high pressure water service, upon which a fire-hose can be at once adapted.

In the frontispiece illustration of this book a block of four laboratory benches in the Nottingham University College is shown; two of these are presented to the observer, and are therefore visible in detail. It is convenient to arrange the benches in this way, since it enables the washing sinks and water taps to be placed at the end of the bench and yet to be accessible to each student: this prevents the surface of the bench from being wetted and soiled when apparatus is being washed.

The sinks are of stoneware, with half-inch outlet pipes unprotected by grids: these pipes discharge into stoneware pots, the overflow from which into the drain-pipes is through a curved stoneware tube near the top: solid matter passing down the sink-pipe settles in this trap-pot, and is got rid of by taking out the pots and emptying them into a pail every few weeks: floating paper or other light substances which may be carried into the main pipes are removed by flushing them occasionally with high pressure water. Any stoppage in the straight sink-pipe is readily pushed through with a stout cane. Over each sink there are three low pressure taps for washing and for supplying condensers, and two high pressure taps for aspirators.

Lead-covered sinks lessen the number of breakages caused by dropping apparatus while it is being washed, but they are easily corroded by acids and are less cleanly: a wooden grid laid on the bottom of a stoneware sink lessens the risk of breakage.

Each bench is provided with an aperture leading to a strong down draught, and into this aperture a copper hood can be fitted, which serves to carry away acid vapours or badly smelling gases. This aperture is stopped with a wooden plug when the hood is not in use, in order to improve the draught on other benches. Copper hoods, which are occasionally cleansed and painted with Brunswick black, answer well: but fixed small earthenware hoods may be more convenient. A glazed draught hood extending along the top of the centre shelf rack of the bench has been found to answer well: all heating and evaporation of liquids is then carried out on a raised shelf under this hood.

The laboratory bench itself is made of stout American walnut wood, the whole of which is stopped and varnished with the exception of the working surface of the bench, which should be well saturated with oil and then rubbed up. This working surface is preserved by being occasionally thoroughly washed, dried and oiled. Less durable and expensive wood covered with sheet lead has been found to serve well for a bench top.

The bench may be 3 feet in height with a depth of 25 inches: and a gangway of 6 feet between the rows of benches is not too broad for convenience of passage.

Two gas taps are fixed on each bench for supplying Bunsen burners, and one lighting burner is provided at a convenient height for illuminating the bench at night. A space of 7 inches between the reagent shelves will be found suitable for ordinary bottles.

The working bench is supplied with two lockers, each of which has two drawers above it. In order to lessen the number of locks, which are liable to rust in the laboratory atmosphere, a flat iron bar is fastened upon a staple by a padlock in front of the two drawers and locker. By this arrangement of lockers two students are enabled to use the same bench at different times. Each locker should have a different key, but all the locks are under the control of master keys kept by the attendant and teachers. The drawers should be about 3 inches in depth, and are conveniently divided by wooden strips: a shelf in the locker should not extend more than half across, so as to leave standing room for tall apparatus.

Besides the ordinary working benches, there should be at least one long bench well supplied with gas and water taps and with overflow pipes: this serves for making up solutions, for distillations and for other large operations. There should be a long bench made of a slate slab, and covered at a height of several feet with a metal hood, under which is a good draught outlet: this bench must be supplied with a half inch gas pipe and taps, and with water taps and overflows: it serves to support water-baths, air-baths, combustion furnaces and gas furnaces of all kinds.

A blowpipe table, the top of which is edged round and is covered with sheet lead or zinc, should be provided: this must be well supplied with gas taps for gas blowpipes and luminous burners for bending glass; and two or more Fletcher's bellows should stand beneath it. It is also well, if possible, to have a water-blowing apparatus near at hand to supply a blast to the blowpipes for long operations.

Opening into the general laboratory there should be at least two separate apartments, one serving for stores of chemicals and apparatus, and the other for a reference library and for delicate metal apparatus, such as chemical balances, microscopes, spectroscopes and polariscopes. This apparatus, more especially the chemical balances, should stand on a broad shelf, which is at a convenient height for sitting; the shelf is supported on iron brackets from the wall, so as to be secure against disturbance from the vibrations of the floor. The spectroscope and polariscope should have draw-curtains of black lining material so arranged that when the instruments are in use light from outside may be pretty well excluded. A large central table should also be provided for use in writing and reading. Special low tables, with a raised flange for gas apparatus and other experiments involving the use of mercury, are necessary: and to provide against the loss of mercury spilt from these tables, it will be well to have the floor covered with linoleum.

LIST OF BENCH APPARATUS.

Detailed description of apparatus is rendered unnecessary by the fully illustrated and descriptive price lists now furnished by the apparatus sellers.

531. A list of the apparatus which should be kept in each bench locker will be found facing page 1: it is repeated here with details of dimensions and other matters. In Section I. of the book directions are given for preparing and fitting apparatus. The Bunsen burner with its india-rubber tube may be left attached to the gas tap on the bench; all the other apparatus should be locked up in the bench locker when not in use. Most of this apparatus is seen in the Frontispiece illustration.

1 Bunsen burner (1), about 5½ inches in height, with ⅜-inch tube, and means of closing the air-holes (1).
1 Rose top to fit the burner.
1 Fletcher's argand Bunsen burner, ¾ inch size, is convenient for boiling the wash-bottle.
1 Piece of red or black india-rubber tubing 5/16 inch in internal diameter and 16 inches long, to supply gas to the burner.
1 Test-tube stand with twelve holes, two of which are at least 1 inch across, and with no draining pegs.
1 Test-tube brush (11), or a cleaner with flat india-rubber end.
12 Test-tubes, 5 inches long, ⅝ inch in internal diameter.
2 Boiling tubes, 6 inches long, 1 inch in internal diameter.
2 Round glass plates, ground on one side, 3 inches across.
2 Berlin porcelain evaporating dishes with spouts, glazed inside and out, and 3 inches in diameter.
2 Watch glasses, 2 inches across.
1 Conical flask of 4 ounces capacity.
1 Wedgewood mortar, 4 inches across, pestle with wooden handle.
1 Galvanised iron tripod stand, 7 inches in height, with round top 4 inches across; these dimensions are suitable to the above burner.
1 Square of coarse iron wire-gauze, 5 inches in the side; best with the corners clipped off.
A square of asbestos millboard may be used instead of the gauze.
3 Plain glass funnels, two of them 2½ inches across, and one 2 inches across.
3 Beakers without lips, wide form, of 1, 2, and 3 ounces capacity.
3 Glass rods, round at the ends, 7, 6, and 3 inches in length.
1 Piece of platinum foil, 1 inch by 1½ inch.
2 Pieces of mounted platinum wire (7), each 2 inches long and about as stout as an ordinary sewing needle.
1 Blowpipe, Black's japanned tin.
1 Pipe-clay triangle, 2 inches along its side.
1 Wash-bottle (10), made by fitting a conical 18 ounce flask, which is at least 1 inch across the inside of the neck.
1 Retort-stand, upright rod 16 inches, foot 6 by 3½ inches, galvanised iron, with 3 rings of brass or gun-metal, the largest 3 inches across.
1 Wooden filter-stand (page 48), rod 12 inches high, two rings on one boss, each 2¼ inches across, foot 5 inches by 8 by ½ inch.
1 Pair of polished brass crucible tongs, 6 inches long.
1 Small horn spatula, 3½ inches long.
1 Wicker oval draining basket, 10 inches by 8, and 4 inches deep.
Cut filter papers 4½, 3½, and 2¾ inches across.

LIST OF SPECIAL APPARATUS FOR SECTION II.

532. This apparatus need not be supplied to each student, but several sets should be kept in an accessible position in the laboratory, whence they may be fetched when required: this apparatus should never be kept in the bench lockers. An asterisk indicates that the apparatus is also required for chemical analysis.

*1 Nest of the three or four of the smallest sizes of brass cork-borers.
*1 Triangular file, without wooden handle.
*1 Thin round file, without wooden handle.
* Several lengths and pieces of hard glass tubing about ⅛ inch internal diameter.
*1 Gross of ignition tubes 3 inches long, ½ inch across.
* Corks as free as possible from holes and cracks, sizes varying from ⅜ to ¾ inch across.
1 Small metal clamp in a boss which fits on the retort stand (*see* figs. 15, 37 on pages 18 and 64).
1 Stoppered bell-jar of 16 ounces capacity.
4 Glass cylinders on feet, with ground edge at top (*see* fig. page 20), 8 inches high, 1¾ inches across.
4 Similar cylinders, 6 inches high, 1½ inches across.
1 Round brown stoneware trough, 12 inches across, 5 inches deep.
1 Metal deflagrating spoon, with brass cap 2½ inches across.
2 Two-necked Woulffe's bottles of 8 ounce capacity.
2 Two-necked Woulffe's bottles of 4 ounce capacity.
1 Tubulated retort of 6 ounces capacity.
2 Thistle funnels, 8 inches long.
1 Clock glass, about 4 inches across.
4 Pieces of red or black india-rubber tubing, $\frac{3}{16}$ inch in internal diameter and 1½ inches long.
* A good supply of wooden spills, or thin strips of wood.
* A good supply of wax tapers, about 11 inches long.
A small piece of thin candle.
1 Tube (fig. 20*a*, page 26), 6 inches long, filled with small pieces of calcium chloride.
4 Strips of sheet lead, 6 inches long, ½ inch broad.

LIST OF APPARATUS FOR GENERAL USE IN ANALYSIS.

533. The following apparatus is kept in the laboratory for the general use of students who are doing chemical analysis.

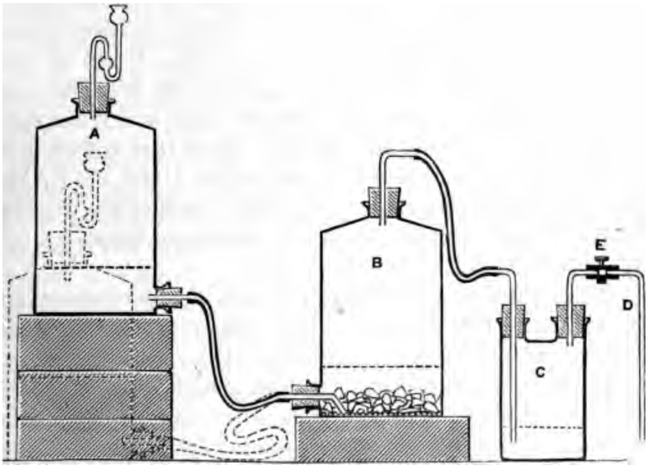
One set will suffice for about twelve students. Each article should have its own place in the laboratory, and should be returned to it immediately after use, since it may be required by other students. Apparatus in (532), which is marked with an asterisk, is also required for analysis.

1 Spirit lamp, 4 ounces in capacity, with earthenware wick-holder and ground glass cap.
1 Charcoal borer of conical form.
4 Tin filter-dryers.
4 Berlin porcelain crucibles, 1½ inch across, with covers.
1 Iron mortar 8 inches across, and pestle.
1 Fletcher's foot-bellows, No. 5; and blowpipe table covered with sheet zinc or lead.
1 Fletcher's Bunsen burner, with central blast, and two taps.
1 Fletcher's solid flame burner, with tripod for boiling large dishes.
1 Fletcher's injector furnace for strongly heating large crucibles.
1 Microscope, with at least a ¼ inch object glass.
1 Spectroscope, Bunsen's table form.
1 Spectroscope, direct vision, pocket form.
1 Indigo prism, stoppered. This is nearly filled with solution of indigo in strong sulphuric acid, the dry stopper is inserted into the dry neck and securely fastened down with fine copper wire: melted paraffin wax is then run over the stopper and neck. The indigo solution is made by mixing commercial sulphindigotic acid with about ten times its measure of strong sulphuric acid, letting settle for several days, and decanting into the prism.
1 Agate mortar 3 inches across, and pestle: which is used for reducing to fine powder hard substances which have been already broken into minute fragments; this must be done by trituration, and blows are on no account to be struck with the pestle.
2 Small leaden cups, about 1½ inches across and ¾ inch deep (296). These are made by beating sheet lead into shape.
2 Copper water-baths, 5 inches across, with four diminishing rings. One large bath with several openings may replace these. The baths when in use should be about two-thirds filled with water, and the flame is adjusted so that the water is kept just boiling. Evaporating basins placed on the bath cannot be heated above 100° C.
2 Pairs of conical flasks fitted as is shown in fig. 41 (page 118).
2 Tubulated flasks, 4 ounces in capacity, see fig. 44 (page 138).
A box of assorted pieces of wood charcoal, as free as possible from cracks and from bark.

SULPHURETTED HYDROGEN APPARATUS.

534. Sulphuretted hydrogen gas is constantly required by the analyst, and should be readily obtainable at any time in a constant stream: arrangements must be made for preventing this badly smelling gas from polluting the atmosphere of the laboratory to any large extent. Both these objects, together with economy of materials, are better secured by the use of a large apparatus in common by many students than by the employment of a small private apparatus by each student.

FIG. 45.



The gas is most readily obtained by the action of somewhat diluted cold commercial hydrochloric acid upon fragments of ferrous sulphide. The apparatus in which the gas is prepared has assumed many forms; the one figured in section and described below is recommended by its simplicity, efficiency and cheapness.

Two large tubulated bottles, A and B, each of at least two quarts capacity, are fitted as is shown in (fig. 45); A and B being connected one to another by a piece of broad india-rubber tubing, and B communicating with the wash-bottle C by narrow rubber tubing: the stoppers used should also be of rubber, as these are more durable and gas-tight than ordinary corks. The delivery tube D is connected with the washing-bottle by means of an india-rubber joint, which can be partially or entirely closed by means of a screw clamp E.

In charging the apparatus, B is laid on its side and lumps of ferrous sulphide are introduced until the bottle is full: the washing-bottle C is then partly filled with water, and the three bottles are connected; the clamp E having been closed, A is about half filled with a mixture in equal measures of strong commercial hydrochloric acid and water.

When the gas is required A is raised on a suitable stand and the clamp E is gradually opened, until a regular stream of bubbles passes through the water in C. A part of the acid will run into B and evolve H_2S from the FeS, and the gas will be forced out by the pressure of the acid remaining in A. Before escaping through the tube D it is washed free from small drops of liquid containing HCl and $FeCl_2$, by passing through the water in C. The levels of the liquids in the bottles while the gas is being produced are shown by the horizontal dotted lines.

When the gas is no longer required, the screw-clamp at E is gradually tightened, until the stream of bubbles through the water in C is arrested; the gas accumulating in B will then force the acid out of B back into A, and if the glass tube at the bottom of B is bent as is shown in the figure, the acid will be almost completely expelled. This will cause the production of the gas to cease, but it will recommence immediately on opening the clamp E.

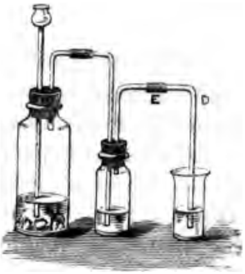
During the hours of laboratory work A should remain raised on its stand, and the apparatus is then always ready for use: at other times A is placed down in the position represented in dotted outline in the figure. B should be permanently somewhat raised to facilitate the outflow of the acid when A is lowered. The escape of H_2S dissolved in the acid is much lessened in amount by fitting a funnel containing water or glycerine in the stopper at the top of A.

When the acid is spent and no longer evolves the gas from the FeS, it is emptied away and replaced by fresh: lumps of FeS are also occasionally filled into B.

The apparatus should stand in a closet, lined with glazed white tiles, and furnished with a good draught; the bottom of the cupboard should be covered with sheet lead, and furnished with a drain-pipe for the spent acid, and a gas jet should be provided for lighting it at night. This closet must not be used for any other purpose. Arrangements should be made for a large opening in front for recharging the apparatus, but a small door only should be used for introducing the solutions through which the gas has to be passed. If the front of the cupboard is opened by means of a glazed sliding sash, a frame containing pairs of glazed hinged doors, each about six inches by eight, may be introduced into the grooves beneath the sash; these doors enable the apparatus to be used with the least possible opening of the closet. Such an apparatus should serve twelve or more students, according to circumstances.

An apparatus for private use may be fitted as shown in fig. 46. The larger bottle contains pieces of ferrous sulphide upon which HCl, diluted with an equal measure of water, is poured through the thistle-funnel: the gas passes through a little water in the smaller bottle, and thence into the solution to be saturated. The use of a small apparatus by each student in a large laboratory is not to be recommended, since it causes unnecessary waste of time and materials, and does not permit of satisfactory control of the stream of gas which is evolved.

FIG. 46.



Passing sulphuretted hydrogen. — Each student should keep a bent glass tube D (figs. 45, 46), which may be fitted upon the sulphuretted hydrogen apparatus, and serves to conduct the gas into a solution. When a liquid is to be saturated with the gas, the end of this tube should be passed down *to the bottom* of the vessel containing the liquid, and a regular stream of gas is then caused to bubble through the liquid by gradually opening the clamp E (fig. 45), or by introducing a sufficient quantity of acid into the larger bottle shown in (fig. 46).

To avoid unnecessary waste of gas, the liquid should be of sufficient depth to absorb much of the gas, and the bubbles should not pass too fast to be readily counted. The passage of the gas should be continued for four or five minutes, if the quantity of metal to be precipitated is large: and the liquid must smell strongly of the gas after the air above it has been blown out. The tube D must be thoroughly cleansed immediately after use.

It will be found convenient to keep a bottle of distilled water saturated with H_2S: this is prepared by passing a regular stream of the gas through water nearly filling the bottle, until on removing the bottle, closing its mouth tightly with the thumb and shaking it violently, no suction is felt on the thumb, but rather an outward pressure.

DISTILLATION OF WATER: STEAM OVENS.

As has been already shown (25), water ordinarily contains certain solid substances dissolved in it which render it in a chemical sense impure; such water is therefore unfit to be employed for the processes of solution and washing precipitates, since any impurity thus introduced into a substance during analysis would be considered when detected to have been present in the original substance.

The quantity of these impurities present in any particular water-supply will depend upon the nature of the soil with which the water has been in contact before its collection. The water supplied in some districts will be found when subjected to the tests given in remark 41 (552), to be almost perfectly pure; carefully collected rain water will yield little or no indication of dissolved impurity; the water supplied to the majority of laboratories will, however, be found to be unfit for analytical purposes, until it has been freed from the solid substances dissolved in it by being subjected to the process of distillation.

In most towns distilled water can be purchased, but it is preferable to distil all the water required for use, either in the laboratory itself or in its immediate neighbourhood. For this purpose the steam obtained from a steam-boiler or from a copper still heated by a furnace or gas-burner, or from the water-baths and steam-ovens in the laboratory, is condensed by a block tin worm-pipe immersed in a tub through which a constant stream of cold water runs.

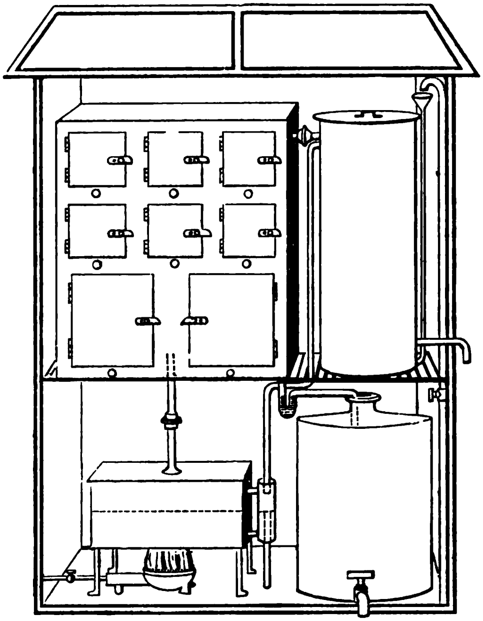
535. *The distillation of water may be advantageously combined with the heating of drying ovens* to a temperature not exceeding 100° C. For this purpose one of the tiled recesses in the laboratory wall is furnished with a glazed projecting hood and with a good draught from the top. Into this recess the arrangement represented in (fig. 47) is fitted: where the copper boiler and a stoneware collecting jar are seen resting on the floor of the recess, whilst the set of double-cased copper ovens and the still-tub are supported on a strong iron grating above.

The steam generated by the powerful Fletcher burner passes from the boiler through a pipe which terminates several inches above the bottom of the outer casing of the ovens. It then makes its way between and around the various ovens, undergoing partial condensation in heating them; and the hot condensed water thus produced flows through a pipe, the opening of which is flush with the bottom of the oven casing, into the stoneware jar, from which hot distilled water may be drawn when the apparatus is at work. This pipe has a U-bend to prevent the escape of steam.

Any steam which is not condensed in heating the ovens passes from the upper part of the oven casing into a block tin worm inclosed in the tall still-tub, and drops as distilled water from the end of the worm pipe into a second stoneware store-jar provided with a tap below. The level of the water inside this jar is shown by a syphon gauge such as that seen in (fig. 48).

The still-tub is supplied with a stream of cold water from a pipe, in which is a control tap, seen at the right hand side of figure; and from the opposite side of the still-tub the heated condensing water flows into a little feeding vessel

FIG. 47.



attached to the side of the boiler: a portion of it serves to keep up the water-level in the boiler to the dotted level in the figure, and the rest flows away into the drain pipes through a central pipe seen in the feeding vessel.

All the connections are made by means of screw union joints, so that the apparatus may be quickly and easily disconnected for removing the incrustation in the boiler and on the condensing worm, and for repairs.

This apparatus has been allowed to run uninterruptedly for several days and nights in succession without any attention, after the gas burner had been lighted and the supply of condensing water had been regulated by the tap.

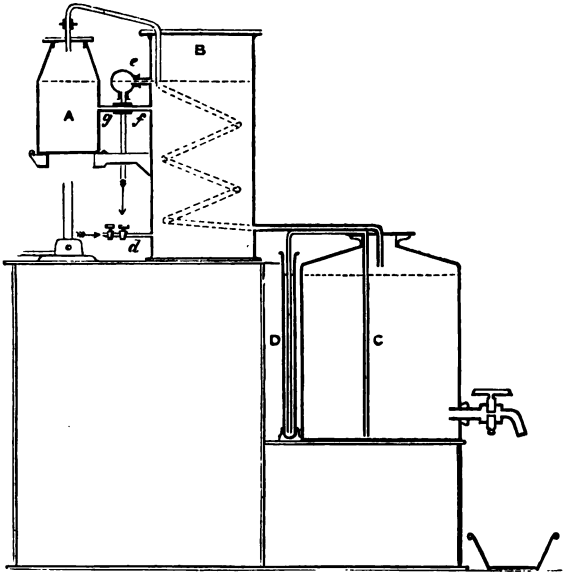
It has been found possible to attain a higher temperature than that usually recorded in a steam-oven, by dispensing with the usual inlet for air through the door, and admitting the air through a narrow copper tube, one end of which is seen immediately beneath each door, the other end opening into the oven flush with its bottom: the pipe itself is thus constantly surrounded with steam, and the air passing through it into the oven enters at a temperature of nearly 100°. There is a small outlet for air provided at the upper part of the back of each oven.

If the processes of distillation and heating the steam ovens are not combined in one apparatus, the constant still figured and described below will be found very serviceable. The sketch in (fig. 48) renders a lengthened description unnecessary: it represents the apparatus in section.

The water in a copper-still (A) is heated by the flame of a Fletcher burner or of a large-sized Bunsen burner, and the steam thus generated passes from the top of the still through a spiral tin tube immersed in water in the condensing tub (B), which is made of copper or galvanised iron; from this condenser the distilled water flows into a large stoneware vessel (C), which is furnished with a tap below from which the water is drawn when required. A long syphon-tube (CD) serves as a gauge to indicate the level of the water inside the jar.

The condensing-tub (B) has three tubes let into the side facing the still; one of these (*d*), placed about an inch above the bottom of the vessel, is the inlet for cold water from a cistern, the water level in which must be higher than that maintained in A and B; this level, marked by the single dotted line, is kept constant by the overflow-tube *e*, which is connected with a pipe leading to a sink or drain. Just below this overflow-tube is a short tube (*f*), which is on a level with another (*g*) opening into the side of the still; when these two tubes are connected by means of an india-rubber joint or a screw union, a supply of warm water from the upper part of the condenser is furnished to the still and keeps the level of water in it constant; a small glass jet drawn out from a piece of glass tubing is fitted into this connecting-tube, and has been found sufficient to prevent an undue circulation of water between the still and the condenser.

Fig. 48.



The still is supported on an iron ring, projecting as a bracket from the condenser. Time is economised by having two taps in the inlet-tube (*d*), or by using two clamps on the india-rubber joint which connects it with the supply-pipe from the cistern; since the flow of water may then be regulated once for all by one tap or clamp, the other being used only for stopping and starting the stream. The copper still should be furnished with a lid screwing upon its mouth for convenience in removing the deposit which collects within, and into this lid should be fastened a brass tube carrying a screw union by which the tin condensing pipe may be fastened steam-tight to the still.

The advantage of this apparatus lies in the fact that the still is constantly supplied with the hottest water from the condenser through the lateral tube, and that when the apparatus has been properly fitted up and the supply adjusted, it requires no attention whatever except lighting and extinguishing the gas under the still, and turning the water supply on and off when the distillation is started and stopped. It is advisable occasionally to remove the incrustation from the interior of the still, and from the supply-tube connecting it with the condensing vessel.

The wooden stand for the still and storing jar is conveniently fitted with shelves and doors to serve as a store cupboard.

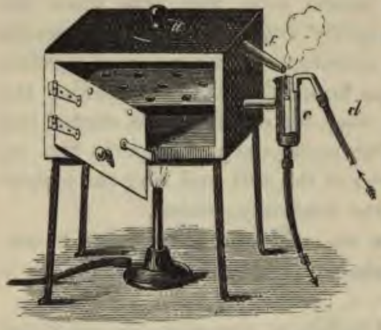
Such a copper-still, 23 inches in circumference below and 8 inches in height, when filled to a height of 4 inches and heated by the largest Bunsen burner, yields two litres of distilled water per hour.

In laboratories which require a larger supply of distilled water than can be furnished by the above apparatus, a copper still of several gallons capacity may be set in masonry and heated by a small furnace fed with coal, coke, or gas. The overflow from the still-tub should then pass into a small cistern the overflow-pipe of which keeps its water surface level with that required in the still: and the still is supplied with hot water by connecting it with the cistern by means of a tube bent downwards in its middle so as to prevent the backward passage of water from the still to the cistern.

The purity of distilled water may be ascertained by the tests described in remark 41 (552).

The steam oven is a small oven of sheet copper, which is used for drying substances at a temperature not exceeding 100° C. If such ovens are not heated by steam from a separate boiler, one or more of them should be heated by boiling water in the space between the oven itself and a copper casing which surrounds it (fig. 49). The outer casing often has a circular opening cut in the top, which serves as a water-bath: this is covered by a lid when not in use. The level of water in the jacket may be maintained constant by a little contrivance shown in section at *c*: a small reservoir communicating by a lateral tube with the space between the oven and its jacket is constantly replenished by water which drops into it from the supply tube (*d*); the overflow of this reservoir is a central tube, the end of which terminates just above the level of the lateral tube. The outlet for steam (*f*) may be turned downwards, so that any water formed by the condensation of the steam drops into the reservoir (*c*), or it may be made to communicate with the worm of the still, when the escaped steam will be condensed into distilled water.

FIG. 49.



The steam-oven should if possible be supplied with distilled water, as the formation of a troublesome incrustation in the interior is thus prevented. A raised vessel of distilled water may be connected with the supply pipe (*d*), and the overflow is collected and occasionally returned to this supply vessel.

Or better still, the escape pipe (*f*) may be turned upwards and connected with a long vertical or oblique tube, which will condense the escaping steam and return it to the oven, thus obviating the necessity of a constant supply of water: in this case the water in the oven should not be boiled too furiously.

RECOVERY OF SILVER AND PLATINUM FROM THEIR RESIDUES.

Any waste scraps of platinum or silver wire or foil, as well as the precipitates obtained in the processes of analysis by the addition of solution of $AgNO_3$ or of $PtCl_4$, should not be thrown down the sinks, but should be placed in two large jars specially reserved for them, and labelled *silver residues* and *platinum residues* respectively. The reagents may be obtained from these residues occasionally by the methods described below.

SILVER RESIDUES.

536. Some HCl is poured into the jar containing the silver residues and is thoroughly mixed up with the liquid: the precipitate is then allowed to subside, and after the decantation of the liquid is thoroughly washed by decantation (31): it may then be treated either by method 1 or 2.

1. The wet precipitate is rinsed into a flat shallow dish and the layer of water is acidified with H_2SO_4: strips of Zn free from Pb are then laid upon the silver precipitate, and the whole is allowed to stand quietly for several hours. The black spongy mass which remains is metallic silver; it is separated from any remaining Zn, and is then thoroughly washed until it is free from H_2SO_4.

This metallic deposit is dissolved by heating it with pure strong HNO_3, previously diluted with its own measure of water. The solution is evaporated to dryness, the solid residue is dissolved in water and once more evaporated to dryness to remove free acid completely. The solid is then dissolved in the proper proportion of water to give reagent 48 (551). Any residue insoluble in HNO_3 is returned to the silver residue bottle.

2. The well-washed precipitate obtained from the silver residue bottle may be filtered off and dried, then mixed with twice its weight of a mixture of K_2CO_3 and Na_2CO_3 in equal proportions. This mixture is transferred to a clay crucible and fused in a furnace: after having been kept in the fused condition for four or five minutes, the crucible is removed and its bottom is tapped several times on a brick to cause the melted globules of Ag to unite. When the crucible is cool it is broken up, the button of Ag at its bottom is removed, well washed, and converted into $AgNO_3$ solution as is described in the preceding paragraph.

PLATINUM RESIDUES.

537. The liquid is shaken up with the precipitate and evaporated to dryness in a porcelain dish, and the residue is heated strongly for some time: when the dish is cool, water is poured upon the residue and boiled with it, then decanted off: solution of oxalic acid is then poured into the dish and evaporated to dryness, and the residue is once more ignited strongly. The residue of metallic Pt is thoroughly washed with boiling water, and is then dissolved by heating it with HCl to which one third its measure of HNO_3 has been added; the solution is evaporated to dryness over a water-bath. HCl is then poured in, and the liquid is once more evaporated to dryness and heated for some time on the water-bath; this residue when dissolved in water forms reagent 28 (551).

Any waste scraps of platinum foil and wire should be carefully preserved, and after having been cleansed by boiling them with HNO_3 and then washing well with water, they may be dissolved in HCl and HNO_3 and solution of $PtCl_4$ prepared as is described above.

CHEMICALS AND REAGENTS.

In the following paragraphs general directions are given for the preparation of reagents and test substances required in the preceding analytical course. Tabulated lists of the names and formulæ of these substances follow, a reference number being attached to each, and certain necessary details concerning its preparation and the tests for its purity being also supplied. Lists of the chemicals required for the experiments in Sections I. and II., and certain special reagents for detecting the rarer elements and organic substances, are also tabulated.

Samples of substances for analysis are given in (558–561), and information concerning atomic weights, thermometric scales and English and French weights and measures, is contained in paragraphs (562–564).

The pure chemicals required in analysis are readily purchased, and it will be found more economical as a rule to buy than to prepare them.

The preparation of the solutions, and the dilution of acids and other liquids should, however, always be performed in the laboratory, since this lessens cost both of purchase and of carriage.

538. Many reagents and test substances must be dissolved or diluted for use. As these processes of solution and dilution must be constantly carried out in a laboratory, it is important that the methods employed should be as simple and rapid in execution as possible, and that the labour should be minimised.

It is also of great importance that the solutions should be of appropriate strength. It is usually advisable that the same liquid should be of different strengths when used for a reagent and as a test solution. In either case a strength can be selected which generally yields the most satisfactory results: if the solution is stronger than this, chemicals are wasted; if it is weaker than this, the reaction is not satisfactorily obtained. Hence it is advisable to prepare these liquids for the student rather than to leave them to be prepared doubtfully or wastefully by him when required: and in order to reduce the labour of preparing solutions to a minimum, it is well to make each in some quantity and to keep it in stock.

The apparatus required for preparing solutions includes—

A common pair of scales, with earthenware slabs and beam below.
A more delicate pair of ordinary scales.
A set of brass weights from 1 kilogram to 1 gram.
A strong glass cylinder to deliver 1 litre, and with graduations for every 5 or 10 cubic centimetres (c.c).
Several large wide-mouthed gallon jars, some of stoneware or earthenware, others of common green glass.
Several large funnels of glass or Wedgwood ware.

A convenient store bottle for liquids is the well-cleansed *Winchester quart*, in which acids and other liquids are constantly supplied to the laboratory.

The process of preparing liquids is naturally divided into the *dilution of liquids* (539), the *solution of solids* (540), and the *solution of gases* (541).

DILUTION OF LIQUIDS.

539. In the lists which follow, the proportions by measure in which liquids are to be mixed with distilled water are stated. Liquids may be mixed in these proportions by measuring them before they are mixed; but the process is often simplified by measuring the height from the bottom of the cylindrical vessel in which the mixture is to be made to the level which the mixture is to reach: this height is then divided in the requisite proportion, and the division is marked by a file or a diamond, by a painted line, or by an india-rubber ring. The liquids are then filled in and thoroughly mixed. The mark once made will always serve for diluting the same liquid again in this vessel.

Thus dilute HCl, No. 2 (550), is made by mixing strong HCl with three times its measure of water. The height of a Winchester-quart bottle to its shoulder was found to be 8 inches; a mark was therefore made on the bottle 2 inches from the bottom, strong acid was poured in until it reached this mark, the bottle was filled to the shoulder with distilled water, then stoppered and shaken, and was thus quickly filled with dilute acid of the required strength.

This method serves for numbers 2, 3, 4, 6, and 7 (550); in the preparation of dilute H_2SO_4 special precautions are necessary, because the strong acid becomes intensely heated by dilution, see remark 1 in paragraph (550).

SOLUTION OF SOLIDS.

540. In the lists which follow there is always placed, in the fourth or fifth column against each solution of a solid, the proportion of solid to water to be used in preparing it. In some of the tables the fourth column gives the number of grams of the solid to be dissolved in the Winchester-quart of water. This number is derived from the proportion of solid to water by considering that an average Winchester-quart bottle filled about an inch above its shoulder holds 2400 c.c. (cubic centimetres) or approximately 2400 grams of distilled water. In a similar manner the weight of water held by any other bottle or vessel may be obtained with sufficient accuracy for this purpose by measuring its capacity in c.c., and counting this number as the weight of water in grams.

Thus the strength given for the AmCl reagent solution, No. 5 (550), is 1 : 8, as is shown in the fifth column; this means that a given weight of water must have dissolved in it one-eighth its weight of solid AmCl: and since a Winchester-quart bottle holds 2400 grams of water, one-eighth of 2400 or 300 grams of AmCl must be dissolved in this water to obtain a solution of the required strength.

Pure solid chemicals are usually sold in the crystalline condition: hence it may be assumed that the crystalline substance is to be used, unless (*a*), which signifies amorphous, is placed against the name in the first column in the table.

If a small quantity of a finely powdered solid has to be dissolved, or if the solid is extremely soluble in water, the solution is often most simply made by introducing the weighed solid at once into the water contained in a stoppered bottle or beaker, and shaking or stirring until solution is completed. This method is successful with No. 10 (550), with Nos. 28, 34, 38, 48, 53, 61, 63, 70, 77, 78, 81 (551, 552), and with many of the substances in (553, 554).

For dissolving large quantities of solids two methods are described below: the former (541) is useful when the solution must be rapidly prepared, but the latter (542) is decidedly preferable for many reasons, and should always be employed if possible.

541. METHOD I.—Weigh out the required quantity of the solid substance, and heat it with distilled water in a large porcelain dish over a Bunsen burner with a rose-top, or over a Fletcher burner; this solution would often crystallise on cooling, hence it should be at once poured into some cold distilled water contained in a large beaker which has been marked to show the quantity of liquid required to fill the store bottle. While pouring off the liquid press a wet glass rod, held vertically, against the lip of the dish, and let the bottom of the dish touch the top of the beaker. These precautions will prevent the liquid from running down outside the beaker.

Dilute this with distilled water to the required volume, mix well by stirring, and if the solution is turbid let it stand covered from the air until it is clear; then pour it off into the store bottle, carefully leaving the sediment behind. If the liquid is wanted in haste, it may be filtered into the bottle: the coarse brown French filter-paper is useful for this purpose, since it is strong and porous: the liquid should be filtered before diluting it to the full extent, as the quantity to be passed through the filter is thus lessened and time is economised.

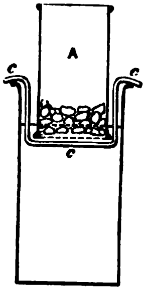
This method is of general application in the solution of solids.

542. METHOD II. depends upon the fact that the specific gravity of water becomes higher as the quantity of solid dissolved in it is increased; hence if the solid is immersed in the water near its surface, a circulation is produced by the constant sinking of the water which has been in contact with the solid and by dissolving some of it has increased in specific gravity, this being replaced by the comparatively lighter liquid from below. This circulation of water over the solid substance gradually dissolves it without the use of heat, and the process requires no attention after it has once been started.

1. The most simple method of applying this principle is to lay the weighed solid upon a piece of dry well washed muslin, then tie this up into a little bag and suspend it in the upper part of the water contained in a jar of common stoneware or earthenware or of thick green glass. A glass rod placed across the mouth of the jar serves to hang the bag upon. The process of solution by this method is very rapid.

2. Or a glass cylinder A (fig. 50) which is open at both ends, such as a broken beaker, flask, or bell-jar, may have one end covered with muslin, which is fastened on by an india-rubber ring and is shown by the lower dotted line: this cylinder with a muslin bottom is supported in the jar by means of two pieces of glass rod or tube, bent as is shown at *c c c*. Distilled water is poured into the jar until it buries the muslin to the depth of about half an inch; the weighed substance is then placed in the cylinder and the whole is allowed to stand until the solid has been dissolved. With large quantities of substance this will usually require several hours, with smaller quantities about twenty minutes or half an hour.

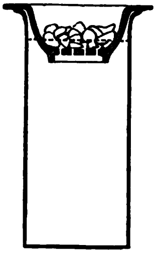
FIG. 50.



3. A convenient substitute for the glass cylinder with muslin bottom is an earthenware colander (fig. 51). If the jar is too large a stoppered bottle of suitable size may be placed in it to raise the water level sufficiently to reach the substance.

The processes of solution and filtration may be performed simultaneously by laying a piece of filter paper on the bottom of the colander, or upon the muslin before fastening it on the cylinder; even the coarse grey paper will however considerably retard the process of solution.

FIG. 51.



Large quantities of substance are most readily dissolved by starting the process over night; the solution will then be completed by the morning. In this case the retardation caused by the filter paper will be immaterial: but the paper is usually unnecessary, since any fine particles which have passed into the water will have settled, and the liquid can therefore be easily decanted from the sediment.

It must be borne in mind that the volume of the water is increased by the solution in it of many salts, and especially of such salts as contain water of crystallisation; hence the quantity of water placed in the jar must be less than the volume of solution required, and the liquid is made up to the required volume after the solution has been effected.

545. *Preparation of saturated solutions.*—A solution is said to be saturated when it contains the largest amount of the substance which the liquid can dissolve at the temperature of the solution. The quantity which can be dissolved usually varies widely with the temperature, and in the case of a solid commonly increases with rise of temperature. Saturation at ordinary atmospheric temperature is always implied below.

The methods described in (542) are very convenient for the preparation of saturated solutions; the solution is known to be saturated when the solid supplied to the upper part of the liquid ceases to be any further dissolved.

In the case of a finely powdered substance, which remains long suspended in water, a saturated solution is also easily obtained by shaking up well with water, allowing the powder to subside and decanting the clear solution. The following solutions will serve as illustrations.

1. *Calcium sulphate solution*, No. 16 (550), is made by filling a Winchester quart up to its shoulder with distilled water, then pouring in some plaster of Paris powder, stoppering the bottle and shaking it well. The liquid will remain milky for a long time, and since the water is thus kept in contact with a large surface of the solid it will become saturated with $CaSO_4$ before the powder has settled. As soon as the liquid has become perfectly clear by settling, the solution is decanted into another bottle for use; the sediment remaining in the first bottle is again shaken up with water and the bottle is set aside; it will thus furnish a fresh supply of the clear solution when required.

2. *Lime water*, No. 57 (552), is prepared by introducing freshly slaked lime into a Winchester quart bottle filled to its shoulder with *tap water*, and proceeding precisely as in the preparation of calcium sulphate solution described in the preceding paragraph.

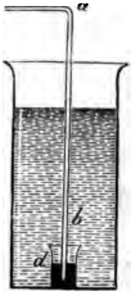
SOLUTION OF GASES.

546. Many gases which are more or less soluble in water, are most conveniently applied as reagents in the dissolved condition. Those which are most frequently used can be readily and cheaply purchased, such as ammonia solution, No. 6 (550), sulphur dioxide solution, No 49 (551), and hydrofluosilicic acid. Of the remaining gaseous solutions mentioned below, sulphuretted hydrogen solution should be kept in readiness, but the gas may be passed through the solution instead; nitrogen tetroxide solution is replaceable by a freshly acidified solution of potassium or sodium nitrite, and as a substitute for chlorine water either bromine water or freshly acidified solution of bleaching powder may be employed.

It will be seen therefore that the preparation of gaseous solutions is by no means indispensable.

All the solutions mentioned below should be prepared in silicon fluoride gas into water. The gas is prepared in a flask fitted as is shown in (fig. 53): into this flask an intimate mixture of 50 grammes of dry, pure, finely powdered fluorspar with 50 grammes of fine white sand is poured; 300 grammes of strong H_2SO_4 are then introduced through the funnel tube, and the acid is mixed with the powder by shaking it round in the flask; the gas evolved on gently heating the flask with the flame from a rose-burner, is made to pass first through the bottle B (fig. 53), which must be empty and dry inside; thence it escapes through a bent tube *a b* (fig. 55), which is fitted upon the outlet tube of B by an india-rubber joint: the end of the tube (*a b*) is made to dip into mercury contained in a small beaker *d* (fig. 55), which stands in a large beaker containing 400 c.c. of water.

FIG. 55.



As soon as the silicon fluoride gas comes into contact with the water after escaping from the mercury, it is decomposed into hydrofluosilicic acid which dissolves in the water, and silica which remains suspended in the water as a gelatinous mass; the silica would very soon close the end of the delivery-tube (*a b*) if it were not protected from contact with water by being immersed in mercury.

As the current of gas slackens the heat is raised, until white fumes of H_2SO_4 begin to appear in the preparation flask; the process is then arrested and the gelatinous silica is separated from the solution by squeezing the liquid through fine muslin and then filtering it if it is not quite clear. The silica may be dried, heated strongly in a porcelain dish and put by in a stoppered bottle as reagent No. 86 (552), for which it serves admirably.

STORE JARS AND BOTTLES.

547. As store jars for large quantities of solid chemicals the broad-necked common green glass jars with flat stoppers, which are used for preserves and confectionery, may be employed. They are very cheap and strong, and present the advantage over stoneware jars that one sees at a glance the quantity of material they contain. The stoppers exclude dust and can be at once made air-tight by fastening a cork ring around their lower part: it is, however, only necessary in the case of very few chemicals to exclude air perfectly. These jars can be purchased in several convenient sizes. Solids are readily removed from them by means of wooden spoons.

The little white glass honey-jars with glass caps serve well for keeping smaller quantities of solids, and can be similarly made air-tight by means of a cork strip. These jars serve for storing the solid powders which are required for analysis in the laboratory (559–561): their necks being of the requisite breadth for inserting a watch glass.

Well made German white glass bottles can be purchased for the reagents and test solutions: these bottles have flat headed stoppers which protect the lip from dust, and each stopper has been ground into the bottle so as to insure its fitting; stopper and bottle are then prevented from permanently parting company by having an identical number etched upon them.

The liquid bench reagents (550) may be kept in narrow-necked bottles of 6 or 8 ounces capacity, and the solids in 4 ounce broad-necked bottles; whilst the special reagents (551, 552) should be in 12 ounce bottles. The test solutions (553, 554) require larger bottles of about 32 ounce capacity: they may be kept in stock in Winchester quart bottles.

The bench reagent solutions may be conveniently kept in stock in an accessible position in the laboratory in large white glass jars with glass taps below: these taps are ground into a tubulure near the bottom of the bottle, and after they have been inserted perfectly dry are fastened by a thick coating of paraffin wax. The plug and seat of the stopper require to be occasionally thoroughly dried and greased. This arrangement of the stock enables each student to fill his own bench bottles when necessary.

Accurately fitting stoppers should not be used in bottles containing solution of KHO or NaHO, as they are certain to be set fast: a loosely fitting stopper or a glass marble placed on the neck obviates this difficulty.

Labelling bottles.

548. Every bottle in use in the laboratory should be labelled as permanently as possible; beginners are much assisted if the label bears not only the name but also the chemical formula of the substance contained in the bottle. It is also extremely convenient to legibly number each bottle in its series, this number serving as a short means of reference and also helping to keep the bottle in its place, so as to enable it to be easily found: this is made more certain still if the edges of the shelves are numbered to correspond with the bottles which should stand on them. The gummed labels should be stored in a dry place.

After the whole surface of the gummed label has been carefully made to adhere to the bottle by pressure with a clean cloth, and it has been allowed to dry thoroughly, it should be brushed rapidly across with an ordinary broad flat and stiff gum brush, which has just been taken out of a little dish of melted paraffin wax. A thin coating of the wax is thus laid on, which should entirely cover the label and project for a short distance beyond its edge upon the glass surface. A little practice will render it possible to lay on an even layer of the wax which does not penetrate and grease the paper. If the paraffin be heated only slightly above its melting point it will set too rapidly to grease the paper.

This paraffin varnish, if properly applied, perfectly resists the action of water, acids and alkalis, and in fact can only be removed or injured by mechanical means, by heat, or by such solvents as ether and turpentine.

Bottles containing the strong mineral acids commonly have ordinary labels etched upon their surface, but this is unnecessary since the paraffined paper label stands perfectly well. Names and formulæ painted on bottles with Brunswick black are also very permanent in the laboratory.

LISTS OF REAGENTS AND CHEMICALS.

549. In the following lists (550–554) will be found each reagent and test substance required in the analytical course. In the first column is a number which may be used for short reference, and which serves to keep the bottle in its place, and to find it when required: in the second column stands the name and in the third the chemical formula of the substance; while the subsequent columns give the strength of the solution and details of its preparation, in cases to which the general methods described in (539–545) do not strictly apply.

An "s" following the reference number in paragraphs (553, 554), means that the substance is required in the solid state as well as in solution, and some of the solid in powder should be placed in a 4 oz. bottle and kept near the solution.

The directions for preparing liquid reagents have been already given in (539–546), it is only necessary to add that in stating the proportions of solid or liquid to water required for the solutions the water always stands last; and that (*m*) signifies proportion *by measure* of liquids, and (*w*) proportion *by weight* of solids to water.

550.—REAGENTS REQUIRE

1. Reference number.	2. Name. [The numbers in brackets refer to remarks correspondingly numbered in the sixth column.]	3. Formula.
Liquids.		
1.	Sulphuric acid (1),	H_2SO_4
2.	Hydrochloric acid (2),	HCl
3.	Nitric acid (3),	HNO_3
4.	Acetic acid (4),	$\bar{H}A$, or $HC_2H_3O_2$
5.	Ammonium chloride (5),	NH_4Cl
6.	Ammonia, or Ammonium hydrate (6)	NH_4HO
7.	Ammonium sulphide (7),	$(NH_4)_2S$
8.	Ammonium carbonate (8),	$(NH_4)_2CO_3$
9.	Ammonium oxalate,	$(NH_4)_2C_2O_4.2H_2O$
10.	Potash, or Potassium hydrate,	KHO
11.	Potassium chromate,	K_2CrO_4
12.	Potassium ferrocyanide,	$K_4FeCy_6.3H_2O$
13.	* Potassium ferricyanide (9),	$K_6Fe_2Cy_{12}$
14.	† Sodium phosphate (10), or Hydric disodic phosphate, or Hydrogen disodium phosphate,	$Na_2HPO_4.12H_2O$
15.	Sodium carbonate,	$Na_2CO_3.10H_2O$ (crystals)
16.	Calcium sulphate,	$CaSO_4$
17.	Barium chloride,	$BaCl_2.2H_2O$
Solids.		
18.	Sodium carbonate (11),	$NaHCO_3$
19.	Borax (12),	$Na_2B_4O_7$
20.	Potassium chlorate,	$KClO_3$
21.	Test papers,	

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* This reagent undergoes decomposition by exposure to light and must not be kept

† Hydrogen sodium ammonium phosphate $(NaAmHPO)_4$ solution serves better for given above for Na_2HPO_4.

FOR EACH BENCH.

4. Weight of solid in grams to be dissolved in one "Winchester" of water.	5. Proportion of solid by weight (w.) and liquid by measure (m.) to water.	6. Remarks. [The small numbers refer to corresponding numbers in the second column.]
. . .	1 : 5 m.	1. Pure strong H_2SO_4 must be poured in the proper proportion into water contained in a large thin beaker or an earthen jar, constantly stirring the water all the time: the right proportions are obtained by measurement of the height of the vessel (539). The hot liquid is cooled by immersing the vessel in cold water, and is then poured into the store bottle. If the acid is pure, no white precipitate ($PbSO_4$) forms during dilution. 2. Must be colourless, and give after dilution no precipitate with either $BaCl_2$ or H_2S. 3. Must give no precipitate after dilution, with $BaCl_2$ or $AgNO_3$ added to separate portions. 4. Must give no precipitate with $BaCl_2$. 5. Must give no precipitate or coloration with Am_2S. 6. Must give only a very slight precipitate with lime water, no coloration with Am_2S, and separate portions acidified with HNO_3 must give no precipitates with $BaCl_2$ and $AgNO_3$. "Liquor ammoniæ fortissima," of 880° specific gravity, is bought. 7. Must be yellow, and give with acids H_2S and a white precipitate of S: it must give no precipitate with solutions of Ca- or Mg-salts. 8. The solid Am_2CO_3 is dissolved by the second method (542) in cold water, but in diluting, one-fourth of the "Winchester" must be filled with strong AmHO. 9. Must give no blue precipitate with Fe_2Cl_6 solution. 10. Must yield no precipitate with AmHO. 11. Dry finely-powdered "bicarbonate of soda." It must, after having been dissolved in excess of HNO_3, give no precipitate with $BaCl_2$ or $AgNO_3$, or $AmHMoO_4$ (277), and if evaporated with excess of HCl must leave no residue insoluble in dilute HCl (291). 12. The borax is best dried by heating in a platinum or porcelain dish until after melting it has again become solid; this solid mass is then finely powdered in a mortar and kept in a stoppered bottle.
... ...	1 : 3 „	
. .	1 : 3 „	
... ...	2 : 1 „	
. 300 . .	1 : 8 w.	
... ...	1 : 3 m.	
. . . .	1 : 1 „	
... 400	1 : 5 w.	
. 100 . .	1 : 24 „	
... 300	1 : 8 „	
. 200 . .	1 : 12 „	
... 200	1 : 12 „	
. 200 . .	1 : 12 „	
... 200	1 : 12 „	
. 600 . .	1 : 4 „	
Saturated solution (545,1)		
. 200 . .	1 12 „	
In small crystals.		
Blue and red litmus and turmeric papers in small strips.		

near a window: it is better to dissolve a fragment of the solid each time it is required.
Precipitation of Mg than does Na_2HPO_4 solution: it is prepared of the strength

551.—REAGENTS FOR GENERAL USE,

Except in large laboratories it will be unnecessary to keep these in the laboratory, may be made up to the right strength as soon as they are

1. Reference number.	2. Name. [The small numbers in brackets refer to remarks correspondingly numbered in the fifth column.]	3. Formula.
25.	Sulphuric acid (17)	H_2SO_4
26.	Hydrochloric acid (2)*	HCl
27.	Nitric acid (3)*	HNO_3
28.	† Platinum chloride	$PtCl_4$
29.	Alcohol, Rectified spirit (18)	C_2H_6O
30.	Tartaric acid (19)	$H_2\overline{T}$ or $H_2C_4H_4O_6$
31.	Acid sodium tartrate, Hydric sodic tartrate (20)	$NaH\overline{T}.H_2O$ $NaHC_4H_4O_6.H_2O$
32.	Slaked lime (21)	$Ca(HO)_2$
33.	Acetic acid (4)*	$H\overline{A}$ or $HC_2H_3O_2$
34.	Cobalt nitrate (22)	$Co(NO_3)_2.6H_2O$
35.	Hydrofluosilicic acid (23)	H_2SiF_6
36.	Sulphuretted hydrogen solution Hydrogen sulphide (24)	H_2S-solution
37.	Oxalic acid	$H_2\overline{O}.2H_2O$ $H_2C_2O_4.2H_2O$
38.	Potassium sulphocyanide	$KCyS$
39.	Potassium nitrate	KNO_3
40.	Lead acetate (25)	$Pb\overline{A}_2.3H_2O$ $Pb(C_2H_3O_2)_2.3H_2O$
41.	Sodium acetate	$Na\overline{A}.3H_2O$ $NaC_2H_3O_2.3H_2O$
42.	Potassium cyanide (26)	KCy or KCN
43.	Bromine-water (27) ‡	Br-water
44.	Stannous chloride (28)	$SnCl_2.2H_2O$
45.	Copper (29)	Cu
46.	Zinc (30)	Zn
47.	Steel (31)	Fe
48.	Silver nitrate	$AgNO_3$
49.	Sulphurous acid	H_2SO_3
50.	Copper sulphate	$CuSO_4.5H_2O$
51.	Magnesium sulphate	$MgSO_4.7H_2O$
52.	Mercuric chloride	$HgCl_2$
53.	Gold chloride	$AuCl_3$

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* See last column on the preceding page.

† Must be used in very small quantity only, being an expensive reagent.

‡ Sodium hypochlorite (Na_2Cl_2O), the "Liquor Sodæ Chlorinatæ" of commerce may be used, but does not keep well.

FOR THE DETECTION OF METALS.

reagents in store in quantity. The 12 oz. bottles in which they are contained empty by the proportions stated below in column 4.

4. Proportion by weight of solid to water: and weight in grams for a 12 oz. bottle in square brackets.
Strong pure.
" "
" "
1 : 30 [15 grams].
Strong.
1 : 10 [45 grams].
Solid.
Strong.
1 : 12 [40 grams].
See (546. 5).
See (546. 1).
1 : 12 [40 grams].
1 : 100 [5 grams].
Solid.
1 : 12 [40 grams].
Solid.
Solid.
1 : 12 [40 grams].
1:100 [5 grams].
See (546. 2).
1 : 12 [40 grams].
1 : 12 [40 grams].
1 : 20 [30 grams].
1 : 30 [15 grams].

5. Remarks.

[The numbers in this column refer to those in brackets in the second column.]

17. Must be colourless and form no brown ring when poured below some $FeSO_4$ solution (247).
18. Strong rectified methylated spirit, which must leave no residue on evaporation, and remain clear on dilution.
19. The solution should be mixed immediately after preparation with several drops of carbolic acid to prevent a vegetable growth forming in it: or better still some crystallised $H_2\overline{T}$ should be dissolved when wanted.
20. Dissolve 10 grams of $H_2\overline{T}$ in 100 c.c.* of water, divide this solution into equal parts, exactly neutralise (34) one part by heating it nearly to boiling and stirring in solid Na_2CO_3 in powder; add to this the other part, cool and dilute to 150 c.c. A few drops of carbolic acid should be added to the solution.
21. Pieces of freshly-burnt lime are placed on a plate, and water is poured upon them until they begin to appear moist on their surface; the superfluous water is then drained off, and as soon as the lime has crumbled to powder, it is placed in a broad-mouthed stoppered bottle.
22. The bottle should be closed with a loosely-fitting india-rubber stopper, perforated, and with a glass tube passed through it and dipping into the solution; when a drop of the solution is required, the upper end of the glass tube is closed by the finger, and a drop is delivered from the opposite end by slightly relaxing the pressure of the finger.
23. The solution must give no precipitate with $Sr(NO_3)_2$ solution.
24. This solution must not darken on adding AmHO.
25. A little $H\overline{A}$ must be added to this solution to make it clear.
26. Solution of KCy is made (1 : 12); it decomposes so readily that the solution is best made immediately before use by heating a small piece of solid KCy with distilled water.
27. A few drops of Br dissolved by well shaking with water.
28. Crystals of $SnCl_2$ should be dissolved by heating them with water containing some HCl; the solution should be kept in a well-stoppered bottle containing pieces of granulated or block tin.
29. In strips cut from thin copper-sheet.
30. In strips or rods, or granulated; it must be proved to be free from As by (181, 182, or 183).
31. Common knitting-needles broken into short lengths, and kept in a bottle containing pieces of quicklime to prevent rusting.

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* c.c. is the contraction for cubic centimetre. See weights and measures (564).

552.—REAGENTS FOR GENERAL USE

Refer to note under

1. Reference number.	2. Name. [The small numbers in brackets refer to corresponding numbers in the fifth column.]	3. Formula.
57.	Lime water	$Ca(HO)_2$
58.	Lead acetate in potash (32)	$Pb\bar{A}_2+KHO$
59.	Potassium dichromate	$K_2Cr_2O_7$
60.	Chlorine water	Cl-solution
61.	Ferric chloride (33)	Fe_2Cl_6
62.	Ferrous sulphate (34)	$FeSO_4.7H_2O$
63.	Potassium iodide	KI
64.	Starch (35)	...
65.	Indigo-solution (36)	
66.	Manganese dioxide (37)	MnO_2
67.	Potassium dichromate (38)	$K_2Cr_2O_7$
68.	Ether (methylated)	$(C_2H_5)_2O$
69.	Carbon disulphide	CS_2
70.	Potassium nitrite (39)	KNO_2
71.	Nitrogen tetroxide solution	N_2O_4-solution
72.	Ammonium molybdate (40)	$(NH_4)HMoO_4$
73.	Calcium fluoride	CaF_2
74.	Potassium disulphate	$KHSO_4$
75.	Microcosmic salt, or Hydric ammonic sodic phosphate (40 a)	$NaAmHPO_4.4H_2O$
76.	Wax, or paraffin	...
77.	Calcium chloride	$CaCl_2.6H_2O$ (crystals)
78.	Potassium chloride	KCl
79.	Marble	$CaCO_3$
30*.	Distilled water (41)	H_2O
81.	Pure sodium hydrate (42), pure soda	$NaHO$
82.	Fusion mixture (43)	$Na_2CO_3+K_2CO_3$
83.	Barium carbonate (44)	$BaCO_3$
84.	Solution of sodium acetate in dilute acetic acid (45)	$Na\bar{A}+H\bar{A}$
85.	Sodium nitrate (46), solid;	$NaNO_3$
86.	Silica, or finely powdered white sand. (See 546. 5.)	SiO_2
87.	Hydrofluoric acid (47)	HF
88.	Zinc sulphate. (See No. 111).	$ZnSO_4.7H_2O$
89.	Calcium carbonate, pure. (See 481.)	$CaCO_3$
90.	Ammonium chloride, pure, in powder. (See 481.)	NH_4Cl
91.	Barium peroxide, in fine powder	BaO_2

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* Nos. 80–91 are certain special

FOR THE DETECTION OF ACID-RADICLES.

the heading of (551).

4. Proportion by weight of solid to water: and weight in grams for a 12 oz. bottle. in square brackets.	5. Remarks. [The numbers in this column refer to corresponding numbers in the second column].
. See (545). . 1 : 24 [20 grams]. ... See (546. 3). . 1 : 24 [20 grams]. ... Solid. . 1 : 60 [8 grams]. ... Solid. ... Solid. . Solid. .. 1 : 24 [20 grams]. . See (546. 4). . In powder. ... In small pieces. . In small crystals. ... Solid in pieces. . 1 : 12 [40 grams]. ... 1 : 12 [40 grams]. . In pieces as large as a pea.	32. To some of the $Pb\bar{A}_2$ solution (No. 40) KHO solution is added until, on warming, the precipitate at first formed is just redissolved: paper dipped into this liquid is a very delicate test for H_2S (230). 33. The solution should not contain any free acid: to remove acid AmHO is added until the further addition of *a single drop* gives a reddish-brown precipitate of $Fe_2H_6O_6$ in the solution. 34. $FeSO_4$ solution rapidly oxidises in the air, hence the $FeSO_4$ should be kept in the solid state as small green crystals, which should not show a yellow coating in any part; these are dissolved when required by crushing and shaking with cold water. 35. Starch solution rapidly changes, it is best therefore to keep the starch as a powder. Starch solution is made by stirring 2 grams of this powder, which has been made into a paste with 10 c.c. of cold water, into 100 c.c. of boiling water and cooling. 36. Made by dissolving indigo-carmine in water. 37. Should be kept in fine powder; it must not evolve Cl or CO_2 when warmed with strong H_2SO_4. 38. In small pieces, or in powder. 39. The solution is prone to change, and should be made in small quantity only: it must evolve copious red fumes when mixed with H_2SO_4. 40. Make the solution with the following proportions:—1 gram of Am_2MoO_4 is dissolved in 12·5 c.c. of strong AmHO which has been previously mixed with an equal quantity of water, the solution is allowed to stand (if necessary) till clear, then poured off into 50 c.c. of strong HNO_3: the liquid will become hot, and should be allowed to stand until it is cool before being used.
	40 *a*. The HNO_3 solution of this salt must yield no precipitate with $AgNO_3$. 41. Must leave no residue on evaporation; in separate portions no precipitates must be caused by $BaCl_2$, $AgNO_3$, or $Am_2C_2O_4$, neither must any precipitate or even dark coloration be produced by addition of Am_2S. 42. The proportion by weight should be 1 : 10 of water; it is best kept in green glass bottles, as it slowly dissolves Pb from white flint glass: The solution must not become dark in colour when mixed with H_2S, nor give a gelatinous precipitate (Al_2Ho_6) when mixed with excess of AmCl solution. 43. Dry finely powdered Na_2CO_3 and K_2CO_3 are intimately mixed in the proportion of 53 : 69 by weight, and kept in a stoppered bottle. 44. Pure powdered $BaCO_3$ is either purchased, or is made by precipitating $BaCl_2$ solution completely with Am_2CO_3 or Na_2CO_3 solution, and washing the precipitate well by decantation. This powder is then mixed with distilled water to the consistency of thin cream. 45. Dissolve 20 grams of $Na\bar{A}$ in 60 c.c. of distilled water, and add to the solution 40 c.c. of strong $H\bar{A}$. 46. Made by neutralising strong hot Na_2CO_3 solution with HNO_3 and evaporating to dryness. Sold also as Chili saltpetre. 47. Purchased and kept in gutta-percha bottles.

reagents entered apart.

553. SOLUTIONS FOR THE

Each bottle should bear on its label the number, name, and formula of tions, containing the powdered solid of each of those substances whose number number, and formula of the substance.

1. Reference number.	2. Name. [The numbers in brackets refer to similarly numbered remarks in the last column.]	3. Formula.
100.s.	Potassium chloride	KCl
101.s.	Ammonium chloride	$AmCl$
102.s.	Sodium chloride	$NaCl$
103.s.	Magnesium sulphate	$MgSO_4.7H_2O$
104.	Barium chloride	$BaCl_2.2H_2O$
105.	Strontium nitrate	$Sr(NO_3)_2.4H_2O$
106.	Calcium chloride	$CaCl_2.6H_2O$
107.s.	Alum, or Aluminium potassium sulphate	$AlK(SO_4)_2.12H_2O$
108.	Ferric chloride (*a*)*	Fe_2Cl_6
109.s.	Ferrous sulphate (48)	$FeSO_4.7H_2O$
110.s.	Chrome alum, or Chromium potassium sulphate	$CrK(SO_4)_2.12H_2O$
111.s.	Zinc sulphate	$ZnSO_4.7H_2O$
112.s.	Manganese chloride (*a*)	$MnCl_2$
113.s.	Nickel sulphate	$NiSO_4.7H_2O$
114.	Cobalt nitrate	$Co(NO_3)_2.6H_2O$
115.s.	Mercuric chloride	$HgCl_2$
116.s.	Lead acetate (49)	$Pb\overline{A}_2.3H_2O$
117.s.	Bismuth nitrate (50)	$Bi(NO_3)_3.5H_2O$
118.s.	Copper sulphate	$CuSO_4.6H_2O$
119.s.	Cadmium sulphate (*a*)	$CdSO_4.4H_2O$
120.s.	Arsenious oxide (*a*) (51) Solution in dilute HCl	As_2O_3
120.	Arsenious oxide (*a*) (52) Solution in water	
121.s.	Sodium arsenate	$Na_2HAsO_4.12H_2O$
122.s.	Antimonious chloride (53) †	$SbCl_3$
123.s.	Stannous chloride (54)	$SnCl_2.2H_2O$
124.	Stannic chloride (55)	$SnCl_4$
125.	Silver nitrate	$AgNO_3$
126.	Mercurous nitrate (56)	$Hg_2(NO_3)_2.H_2O$

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* *a* signifies that the solid substance is

† Solid Sb_2O_3 or $K(SbO)\overline{T}$ (Tartar emetic)

REACTIONS OF THE METALS.

he substance. Small 4 oz. wide-mouthed bottles should be kept near the solu-
has a small (s) affixed to it; these bottles should also carry on their label the name,

4. Weight of solid in grams to be dissolved in one "Winchester."	5. Proportion by weight of solid to water.
100	1 : 25
200	1 : 12
50	1 : 50
50	1 : 50
25	1 : 100
200	1 : 12
200 (in crystals)	1 : 12
200	1 : 12
25	1 : 100
25	1 : 100
50	1 : 50
25	1 : 100
25	1 : 100
50	1 : 50
50	1 : 50
50	1 : 50
50	1 : 50
25	1 : 100
50	1 : 50
25	1 : 100
10	1 : 250
50	1 : 50
25	1 : 100
25	1 : 100
[25	1 : 100
25	1 : 100

6.

Remarks.

[These numbers correspond to the small reference numbers in brackets in the second column.]

48. The solution must be made acid with a little H_2SO_4, and some clean iron nails kept in it, or better, a small quantity is freshly prepared when required, see No. 62 (552)

49. A little $H\bar{A}$ must be added to this solution to make it clear.

50. 25 grams of $Bi(NO_3)_3$ must be dissolved by heating with 25 c.c. of strong HCl diluted with 25 of water; this solution is cooled and poured into the bottle, and the measure made up by pouring in dilute HCl containing one-twentieth of its volume of strong HCl.

51. Dissolve 10 grams of As_2O_3 by heating it with 50 c.c. of strong HCl mixed with 50 c.c. of water, and dilute to the Winchester-quart.

52. Boil excess of As_2O_3 for several minutes with 2000 cc. of water, and filter into the quart-bottle.‡

53. 28 grains of crystallised $SbCl_3$ are dissolved in 25 c.c. of strong HCl mixed with 25 c.c. of water, and diluted to the quart‡ with HCl mixed with four times its measure of water.

54. Boil 25 grams of $SnCl_2$ with 50 c.c. of strong HCl mixed with 50 c.c. of water, as soon as it has dissolved to a clear solution dilute to the quart‡. Scraps of Sn must be kept in the bottle.

55. Heat 25 grams of $SnCl_2$ with 25 c.c. of strong HCl and 100 c.c. of water; whilst constantly stirring, add $KClO_3$ to the hot solution until the liquid turns yellow and Cl is smelt, boil off the Cl and dilute to the quart‡.

56. Dissolve by warming 25 grams of $Hg_2(NO_3)_2$ with 6 c.c. of strong HNO_3 diluted with 114 c.c. of water, then pour water into this solution until it is diluted to a quart‡. Keep Hg in the bottom of the bottle.

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not in the crystalline condition.

may be used for the blowpipe reaction.

‡ Winchester-quart.

554. SOLUTIONS FOR

Refer to the note at the head of the preceding Table, which is amongst the reagents (550–552) and the solutions for the reaction

1. Reference Number.	2. Name. [The small numbers in brackets refer to the corresponding numbers in the sixth column.]	3. Formula.
130.	Sodium sulphate	$Na_2SO_4.10H_2O$
131.s.	Sodium carbonate (57)	$NaHCO_3$
	Marble (calcium carbonate) (57)	$CaCO_3$
132.s.	Ferrous sulphide (58)	FeS
	Ammonium sulphide (58)	Am_2S
133.s.	Acid sodium sulphite	$NaHSO_3$
134.	Sodium thiosulphate	$Na_2S_2O_3.5H_2O$
135.	Sodium hypochlorite (59)	Na_2Cl_2O
136.s.	Potassium nitrite (*a*)	KNO_2
137.s.	Potassium nitrate	KNO_3
138.s.	Potassium chlorate	$KClO_3$
139.s,	Sodium chloride (60)	$NaCl$
140.s.	Sodium bromide	$NaBr$
141.s.	Potassium iodide	KI
142.	Sodium phosphate	$Na_2HPO_4.12H_2O$
143.	Sodium arsenate (61)	$Na_2HAsO_4.12H_2O$
144.s.	Borax (62)	$Na_2B_4O_7.10H_2O$
145.	Potassium bichromate	$K_2Cr_2O_7$
146.s.	Sodium silicate (63)	Na_2SiO_3
	Silica (white sand)	SiO_2
147.s.	Fluor-spar (calcium fluoride)	CaF_2
148.	Hydrofluosilicic acid (64)	H_2SiF_6
149.s.	Potassium cyanide (65)	KCy
150.	Potassium ferrocyanide	$K_4FeCy_6.3H_2O$
151.	*Potassium ferricyanide	$K_6Fe_2Cy_{12}$
152.	Potassium sulphocyanide	$KCyS$
153.s.	Ammonium oxalate (66)	$Am_2C_2O_4.2H_2O$
154.s.	Tartaric acid (67)	$H_2\overline{T}$
	Sodium tartrate (68)	$Na_2\overline{T}.2H_2O$
155.s.	Sodium acetate (69)	$Na\overline{A}.3H_2O$

* See No. 13 (550).

REACTIONS OF THE ACID-RADICLES.

applicable to this. Many of the solutions in this list are already prepared
metals (553).

4. Weight of solid in grams to be dissolved in one Winchester-quart.	5. Proportion by weight of solid to water.
25	1 : 100
100	1 : 25
100	1 : 25
100	1 : 25
100	1 : 25
50	1 : 50
50	1 : 50
50	1 : 50
12	1 : 200
50	1 : 50
50	1 : 50
25	1 : 100
In fine powder.	
50	1 : 50
25	1 : 100
25	1 : 100
25	1 : 100
100	1 : 25
... ...	1 : 10

6. Remarks.

[The numbers correspond to the small reference numbers in brackets in the second column.]

57. The $NaHCO_3$ is required only in the solid form, and can be taken from the reagent bottle on the working bench, No. 18 (550). The marble is kept in small pieces as large as a pea; it is found in reagent bottle No. 79 (552).

58. The FeS is kept as a solid in small pieces as large as a pea. The Am_2S can be taken from reagent bottle No. 7, on the working bench.

59. Strong "Liquor Sodæ Chlorinatæ," diluted with an equal measure of water.

60. No. 102, paragraph (553).

61. No. 121, paragraph (553).

62. No. 19, paragraph (550).

63. The solution is made by dissolving 100 grams of the thick syrup, sold as "soluble glass," in water, and diluting to the Winchester-quart. The solid substance to be used is sand finely ground.

64. No. 35, paragraph (551).

65. No. 42, paragraph (551). The solution decomposes by keeping, a little solid is dissolved when required.

66. No. 9, paragraph (550).

67. No. 30, paragraph (551).

68. 123 grams of $H_2\overline{T}$ are dissolved by heat in 500 c.c. of water, the hot solution is exactly neutralised with solid Na_2CO_3, and then diluted to a Winchester-quart.

69. A little of the solid substance (see No. 41, (551). is dissolved, when required, in water.

555. TEST SUBSTANCES AND REAGENTS FOR THE RARER ELEMENTS (489), AND ORGANIC SUBSTANCES (504).

Only those substances and reagents are entered here which do not occur in the preceding list. A small quantity of solution is made when required.

TEST SUBSTANCES.		REAGENTS.	
Thallium sulphate,	Tl_2SO_4	Mercuric cyanide, solution,	$HgCy_2$
Sodium tungstate,	Na_2WO_4	Potassium bicarbonate, solution,	$KHCO_3$
Palladium chloride,	$PdCl_2$	Potassium bisulphate, solid,	$KHSO_4$
Ammonium molybdate,	Am_2MoO_4	Tin, granulated,	Sn
Iron selenide,	FeSe	Hydrogen peroxide, dilute acid solution,	H_2O_2
Potassium selenate,	K_2SeO_4	Barium peroxide, in fine powder,	BaO_2
Lead telluride,	PbTe	Calcium sulphate, in fine powder,	$CaSO_4$
Potassium tellurate,	K_2TeO_4		
Uranium nitrate,	$UO_2(NO_3)_2$		
Indium sulphate,	$In_2(SO_4)_3.9H_2O$		
Beryllium sulphate,	$Be_2(SO_4)_3$		
Titanium oxide	TiO_2		
Sodium vanadate,	$NaVO_3$		
Lithium chloride,	LiCl		
Rubidium chloride,	RbCl		
Cæsium chloride,	CsCl		
Sodium formate,	$NaHCO_2$	Mercuric nitrate, neutral solution,	$Hg(NO_3)_2$
Sodium citrate,	$Na_3C_6H_5O_7$	Gelatin, isinglass,	——
Ammonium succinate,	$Am_2C_4H_4O_4$	Quicklime, in powder,	CaO
Ammonium benzoate,	$AmC_7H_5O_2$	Tannic acid, powder,	$C_{27}H_{22}O_{17}$
Tannic acid,	$C_{14}H_{10}O_9$	Potassium bicarbonate, solution,	$KHCO_3$
Gallic acid,	$C_7H_6O_5$	Iodic acid, solution (1 : 15),	HIO_3
Ammonium urate,	$AmC_5H_3N_4O_3$	Iodine, in small fragments.	——
Salicylic acid,	$C_7H_6O_3$	Yeast, from a brewer.	——
Meconic acid,	$C_7H_4O_7$		
Cinchonine sulphate,	——		
Quinine sulphate,	——		
Morphine hydrochlorate,	——		
Strychnine,	$C_{21}H_{22}N_2O_2$		
Narcotine,	$C_{22}H_{23}NO_7$		
Brucine,	$C_{23}H_{26}N_2O_4$		
Starch powder,	$(C_6H_{10}O_5)n$		
Glucose,	$(C_6H_{12}O_6)_n$		
Urea,	CH_4N_2O		
Albumen	——		

556. CHEMICALS REQUIRED FOR SECTION II.

In the following list those substances which are used for Section II. only are marked by letters of the alphabet. The bottles containing these substances should be arranged apart in alphabetical order. Against others a number is placed; this indicates that the substance is used for analytical purposes, and will therefore be found, together with any necessary descriptive remarks, in one of the preceding lists (550–554).

All numbers below 22 refer to bottles standing on the shelves above the working bench. If a substance is required for more than one experiment, its entry is not repeated.

Letter or number of reference.	Name.	Formula.	Remarks.
A.	Mercuric oxide	HgO	The orange-coloured *precipitated* oxide is best.
20.	Potassium chlorate	$KClO_3$	—
B.	Manganese dioxide	MnO_2	Commercial oxide in powder.
C.	Wood charcoal	C	In pieces as large as a hazel-nut.
57.	Lime water	$Ca(HO)_2$	—
D.	Sulphur	S	Pieces of roll sulphur as large as a pea.
21.	Litmus and turmeric papers	—	—
59.	Potassium dichromate	$K_2Cr_2O_7$	—
E.	Phosphorus	P	Pieces as large as half a pea, kept in a stoppered bottle under water.
63.	Potassium iodide	KI	—
64.	Starch powder	—	—
F.	Granulated zinc	Zn	Not necessarily pure.
26.	Hydrochloric acid	HCl	Strong, pure.
G.	Calcium chloride	$CaCl_2$	Fused solid, in small pieces.
H.	Marble	$CaCO_3$	In small pieces.
I.	Hydrochloric acid	HCl	Strong, commercial.
J.	Litmus solution	—	—
K.	Copper	Cu	Turnings, clippings, or filings.
L.	Nitric acid	HNO_3	Strong, commercial.
M.	Ammonium chloride	NH_4Cl	Solid, commercial.
32.	Slaked lime	$Ca(HO)_2$	—
N.	Strong ammonia solution	NH_4OH	Liquor ammoniæ fortissima.
O.	Oxalic acid	$H_2C_2O_4.2H_2O$	Solid, in crystals.
P.	Sulphuric acid	H_2SO_4	Strong, commercial.
Q.	Sodium hydrate	$NaHO$	Strong solution in water.
R.	Manganese dioxide	MnO_2	In small pieces.
S.	Oil of turpentine	—	—
T.	Dutch metal	—	Leaf from a small book.
U.	"Turkey red"	—	In strips.
V.	Sodium chloride	$NaCl$	In pieces as large as a hazel nut broken from a lump of rock salt.
3.	Nitric acid	HNO_3	
48.	Silver nitrate	$AgNO_3$	
W.	Ammonium nitrate	NH_4NO_3	Solid, in small pieces.
6.	Ammonium hydrate	NH_4HO	
9.	Ammonium oxalate	$(NH_4)_2C_2O_4$	
17.	Barium chloride	$BaCl_2$	
7.	Ammonium sulphide	$(NH_4)_2S$	
39.	Potassium nitrate	KNO_3	

557. CHEMICALS REQUIRED FOR SECTION III.

All the substances, with those exceptions* only which are named in this list, are required either for analytical purposes and are tabulated in paragraphs (550–554), where they may be found by the number placed in the first column; or they are already entered on the list of substances required for experiments on the gases (556), where they may be found by the reference letter in the first column. A letter (s) affixed to the number of reference indicates that the substance is required in the solid condition, see note at heading of (553).

Number or letter of reference.	Name.	Formula.
39.	Potassium nitrate	KNO_3.
118.s.	Copper sulphate	$CuSO_4.6H_2O$.
79.	Marble	$CaCO_3$.
2.	Hydrochloric acid	HCl.
45.	Copper	Cu.
3.	Nitric acid	HNO_3.
17.	Barium chloride	$BaCl_2.2H_2O$.
8.	Ammonium carbonate	Am_2CO_3.
——	Filter paper	In sheet, or cut.
1.	Sulphuric acid	H_2SO_4.
A.	Mercuric oxide	HgO.
M.	Ammonium chloride	NH_4Cl.
100.s.	Potassium chloride	KCl.
18.	Sodium carbonate	Na_2CO_3.
B.	Manganese dioxide	MnO_2.
19.	Borax	$Na_2B_4O_7$.
62.s.	Ferrous sulphate	$FeSO_4.7H_2O$.
102.	Sodium chloride	$NaCl$.
——	Wood charcoal	C.
116.s.	Lead acetate	$Pb\bar{A}_2.3H_2O$.
118.s.	Copper sulphate	$CuSO_4.6H_2O$.
6.	Ammonium hydrate	NH_4HO.
21.	Litmus paper	——
	Turmeric paper	——
——	Zinc oxide	ZnO.

---

* The exceptions are filter paper (531), wood charcoal (533), and zinc oxide.

SUBSTANCES TO BE GIVEN FOR ANALYSIS BY THE PRECEDING TABLES.

It must be understood that the substances mentioned below are merely brought forward as examples of what may be given to the student for analysis, the teacher will use his own judgment in adopting and extending the list.

SUBSTANCES FOR ANALYSIS WHILST TRYING THROUGH THE REACTIONS FOR METALS AND ACID-RADICLES.

558. After working through the reactions for a group of metals or acid-radicles, a few unknown substances containing only one member of the group are tested and the number of members present is gradually increased in those subsequently given. It is well to keep these substances in the dissolved state (553, 554), since they are intended mainly to afford practice in separation and detection according to the group-tables, and the time spent by the student in preparing the solution would therefore be wasted. Occasionally, however, a solid substance may be given in which the metal or acid-radicle present is to be detected by blowpipe tests or by other tests made on the solid substance. The following will serve as examples :—

Group V.	*Group IV.*	*Group III.b.*
Solution of $NaCl$	Solution of $BaCl_2.2H_2O$	Solution of $ZnSO_4.7H_2O$
" " NH_4Cl	" " $Sr(NO_3)_2.4H_2O$	" " $MnCl_2$
" " KCl	" " $CaCl_2.6H_2O$	" " $NiSO_4.7H_2O$
" " $NaCl+KCl$	" " $Sr(NO_3)_2.4H_2O+CaCl_2.6H_2O$	" " $Co(NO_3)_2.6H_2O$
" " $NaCl+NH_4Cl+KCl$	" " $BaCl_2.2H_2O+Sr(NO_3)_2.4H_2O+CaCl_2.6H_2O$	" " $MnCl_2+ZnSO_4.7H_2O$
" " $NaCl+NH_4Cl$	" " $BaCl_2.2H_2O+Sr(NO_3)_2.4H_2O$	" " $Co(NO_3)_2.6H_2O+NiSO_4.7H_2O+ZnSO_4.7H_2O$
" " $MgSO_4.7H_2O+NaCl+KCl$		&c., &c., &c.
Solids : $NaCl$, NH_4Cl, KCl, $AmCl+NaCl$, &c.		*Solids* : $MnCl_2$, $NiSO_4.7H_2O$, $Co(NO_3)_2.6H_2O$. $ZnSO_4.7H_2O$, &c.

SUBSTANCES FOR ANALYSIS BY THE DIRECTIONS CONTAINED IN SECTION V.

559. Any one of the solutions named in paragraphs (553, 554) may be given for analysis by Section V., some of them being neutral, some acid, and others alkaline in reaction. The following more difficult solutions with acid or alkaline reaction may also be added to the list:—

Acid.	*Alkaline.*
$Ca_3(PO_4)_2$, "Bone-ash" dissolved in dilute HCl.	$KSbO_3$ dissolved in water.
BaC_2O_4 dissolved in dilute HCl.	Na_2SiO_3, solution of "soluble glass."
$Mg(BO_2)_2$[*] " " HCl.	Na_2SnO_3, "preparing salt" of the dyer.
$BaCrO_4$ " " HCl.	

Any of the solid salts which were dissolved in order to prepare the solutions in paragraphs (553, 554) may be given for analysis as solid substances by (331 *et seq.*).

As examples of substances possessing metallic appearance (366), the following may be mentioned as suitable for analysis:—

Zn, in pieces or filings.	NiAs, Kupfernickel.
Pb " "	Graphite, or Black lead.
FeS_2, Iron Pyrites.	Iron filings.

As insoluble substances which require to be examined by (367 *et seq.*) the following may be given for analysis:—

$BaSO_4$.	CaF_2, Fluor spar.
$PbSO_4$.	$FeCr_2O_4$, Chrome Iron Ore.
AgCl.	S, as pieces of roll sulphur.
SnO_2, Tinstone.	C, as powdered wood charcoal or plumbago.

---

[*] Made by mixing hot solutions of $Na_2B_4O_7$ and $Mg(NO_3)_2$.

SOLID SUBSTANCES TO BE ANALYSED BY THE PRELIMINARY EXAMINATION IN SECTION VI.

560. For the Preliminary Examination for Metals (385):—

Simple.			*Complex.*	
NH_4Cl $HgCl_2$ As_2O_3 KNO_3 Cr_2O_3	$Pb(NO_3)_2$ $NaCl$ $BaCl_2.2H_2O$ $Sr(NO_3)_2.4H_2O$ $CuSO_4.6H_2O$ (See note.)	$ZnSO_4.7H_2O$ $MnCl_2$ $Co(NO_3)_2.6H_2O$ $NiSO_4.7H_2O$ SnO_2	$AmCl+NaCl$ $HgCl_2+BaCl_2.2H_2O$ $MnCl_2+KCl$ $NiSO_4.7H_2O+$ $ZnSO_4.7H_2O$	$AlK(SO_4)_2.12HO$ $SnO_2+Sr(NO_3)_2.4H_2O$ $CaCO_3+ZnSO_4.7H_2O$ $Cr_2O_3+MnCl_2+$ $NaCl$

Note.—Best finely powdered, since its colour is then almost destroyed.

For the Preliminary Examination for Acid-radicles (410):—

Simple.			*Complex.*
$CaCO_3$ $Na_2SO_3.7H_2O$ $CaCl_2O$ $NaCl$	FeS KNO_3 $KClO_3$ KI	$Na_2S_2O_3.5H_2O$ KBr $Na\overline{A}$	$CaCO_3+KNO_3$ $CaCl_2O+CaF_2$ Na_2CO_3+KI

A few of the above mentioned solids may then be examined by both Preliminary Examinations for both Metals and Acid-radicles.

SUBSTANCES TO BE ANALYSED BY THE GENERAL COURSE IN SECTION VI.

561. The following lists are so arranged that the analysis of the substances contained in them is more difficult in each column proceeding from left to right, and usually also in proceeding in one and the same column from top to bottom. For examples of Alkaline solutions, see (559).

561. SUBSTANCES FOR ANALYSIS BY THE GENERAL COURSE.

1. Simple soluble substances.	2. Complex soluble substances.	3. Complex soluble substances.
$BaCl_2.2H_2O$ $MnCl_2$ $HgCl_2$ $MgSO_4.7H_2O$. KCl $NiSO_4.7H_2O$ *$CaCO_3$ $Pb(NO_3)_2$ *As_2O_3 Fe_2Cl_6 NH_4Cl $NaCl$ &c., &c.	$CaCl_2.6H_2O+BaCl_2.2H_2O+Sr(NO_3)_2.4H_2O$ $AlK(SO_4)_2.12H_2O+CrK(SO_4)_2.12H_2O$ $MnCl_2+ZnSO_4.7H_2O+CuSO_4.6H_2O$ $Pb(NO_3)_2+HgCl_2+NaCl$ *$As_2O_3+CaCO_3+BaCO_3$ *$ZnO+MgCO_3+KClO_3$ *$MgCO_3+BaCO_3+PbCO_3$ &c., &c.	Fe_2Cl_6 $MnCl_2$ $ZnSO_4.7H_2O$ KCl As_2O_3 $CrK(SO_4)_2$ $12H_2O$ $Co(NO_3)_2.6H_2O$ $Hg_2(NO_3)_2.H_2O$ $Sr(NO_3)_2.4H_2O$ KNO_3 $Bi(NO_3)_3.5H_2O$. &c., &c.

4. Substances yielding a Phosphate precipitate in Group III.	5. Substances partly or entirely insoluble.	
$CaCl_2.6H_2O$ Fe_2Cl_6 $Na_2HPO_4.12H_2O$	$BaSO_4$ SiO_2+S $BaSO_4+CaF_2+SnO_2$	*Metallic substances* (467 *et seq.*) Iron pyrites (FeS_2) Iron filings (Fe) Zinc clippings (Zn) Brass filings (Cu+Zn) German silver (Cu+Zn+Ni) Bronze (Cu+Sn) Type-metal (Sb+Sn+Pb)
$BaCl_2.2H_2O$ $MgCl_2$ Fe_2Cl_6 $Na_2HPO_4.12H_2O$	$BaCl_2.2H_2O$ $AlK(SO_4)_2.12H_2O$ $AgNO_3$	*Silicates* (480)— Fire-clay ($Al_2O_3.2SiO_2$) Brown-clay (do. +Fe) Window-glass (Na, Ca, SiO_2) Flint-glass (Pb, K, SiO_2)
$Co(NO_3)_2.6H_2O$ $CaCl_2.6H_2O$ $MnCl_2$ $CrK(SO_4)_2.12H_2O$ $Na_2HPO_4.12H_2O$ &c., &c.	SiO_2 S C $PbSO_4+BaSO_4$ $PbCrO_4$, ignited.	*Cyanogen Compounds* (482)— Any of the foregoing which has been mixed with KCy, KCyS, $K_4FeCy_6.3H_2O$, $K_6Fe_2Cy_{12}$, $K_6Co_2Cy_{12}$

* To be given in the solid state.

562. LIST OF CHEMICAL ELEMENTS, WITH THEIR SYMBOLS AND ATOMIC WEIGHTS.

The words in brackets are the Latin names of the elements from which the symbols have been derived.

Name.	Symbol.	Atomic Weight.	Name.	Symbol.	Atomic Weight.
Aluminium . .	Al	27·1	Molybdenum . .	Mo	95·7
Antimony (stibium) .	Sb	120·2	Nickel . . .	Ni	58·1
Arsenicum . .	As	75·1	Niobium . .	Nb	94
Barium . . .	Ba	137	Nitrogen . .	N	14
Beryllium . .	Be	9·1	Osmium . .	Os	199
Bismuth . . .	Bi	208	Oxygen . . .	O	16
Boron . . .	B	11	Palladium . .	Pd	106
Bromine . .	Br	80	Phosphorus . .	P	31
Cadmium . .	Cd	112·1	Platinum . .	Pt	194·9
Cæsium . . .	Cs	132·9	Potassium (kalium) .	K	39·1
Calcium . . .	Ca	40·1	Rhodium . .	Ro	104·3
Carbon . . .	C	12	Rubidium . .	Rb	85·5
Cerium . . .	Ce	140·7	Ruthenium . .	Ru	104·5
Chlorine . .	Cl	35·5	Selenium . .	Se	79
Chromium . .	Cr	52·1	Silicon . . .	Si	28·3
Cobalt . . .	Co	59	Silver (argentum) .	Ag	107·9
Copper (cuprum) .	Cu	63·3	Sodium (natrium) .	Na	23
Didymium . .	D	142·5	Strontium . .	Sr	87·6
Erbium . . .	E	166·3	Sulphur . . .	S	32·1
Fluorine . .	F	19	Tantalum . .	Ta	182·6
Gold (aurum) . .	Au	196·6	Tellurium . .	Te	128·2
Hydrogen . .	H	1	Thallium . .	Tl	204·2
Indium . . .	In	113·7	Thorinum . .	Th	232·5
Iodine . . .	I	126·8	Tin (stannum) .	Sn	118
Iridium . . .	Ir	193·1	Titanium . .	Ti	48
Iron (ferrum) . .	Fe	56	Tungsten (wolframium) .	W	184
Lanthanum . .	La	138·3			
Lead (plumbum) .	Pb	207	Uranium . .	U	239
Lithium . . .	L	7	Vanadium . .	V	51·4
Magnesium . .	Mg	24	Yttrium . . .	Y	89·1
Manganese . .	Mn	55	Zinc . . .	Zn	65
Mercury (hydrargyrum) .	Hg	200·2	Zirconium . .	Zr	89·6

563. THERMOMETRIC SCALES.

There are two different thermometric scales in use in this country, the Centigrade and Fahrenheit; the former of these is rapidly becoming universal for scientific purposes. The two scales are mutually convertible by the following formulæ, in which F.° represents a temperature on the Fahrenheit scale, C.° a temperature on the Centigrade scale:—

$$\left.\begin{array}{l} \frac{5}{9}(\mathrm{F.}^\circ - 32) = \mathrm{C.}^\circ \\ \frac{9}{5}\,\mathrm{C.}^\circ + 32 = \mathrm{F.}^\circ \end{array}\right\}$$

The temperatures occasionally referred to in this treatise are given on the Centigrade scale.

WEIGHTS AND MEASURES.

564. The corresponding values of the French and English weights and measures are here given; the use of the French or decimal system is strongly recommended by its extreme simplicity, since the smaller denominations are obtained by taking a tenth, hundredth, thousandth, &c., of the unit chosen, and are designated by the Latin prefixes deci-, centi-, milli-, &c., whilst the higher denominations are 10 times, 100 times, 1000, &c. times the unit, and are named by the Greek prefixes deca-, hecto-, kilo-, &c.; examples of this will be found in the tables given below.

The starting-point of the French system is the "metre" (=39·37 inches); this is the "unit of length." The "unit of measure" is the "litre," which is one cubic decimetre: the "unit of weight" is the gramme, which is the weight of 1 cubic centimetre of distilled water at 4° C.

The chief conveniences arising from the use of this system are:—

1st. That all the different denominations can be written as one, since they are either multiples by ten or are decimal fractions of the unit. Thus 5 decagrams, 3 grams, 4 decigrams, 8 milligrams would be written 53·408 grams.

2nd. That since 1 cubic centimetre of water at 4° C. weighs 1 gram we may obtain the *weight* of water to be used from the *measure* by simply converting the measure into cubic centimetres, the number thus obtained will represent at once the corresponding weight of water in grams. Of course this conversion is strictly accurate only when the water is measured at 4° C., but for ordinary purposes the error introduced when the water is at the temperature of the air is too small to be of any importance in the preparation of solutions.

The weights and measures most frequently used for chemical purposes are the gram, the millimetre (m.m.), the litre, and the cubic centimetre (c.c.) which is $\frac{1}{1000}$ of a litre.

ENGLISH WEIGHTS AND MEASURES.

APOTHECARIES WEIGHT.

lb.		oz.		drms.		scruples.		grains.
1	=	12	=	96	=	288	=	5760
		1	=	8	=	24	=	480
				1	=	3	=	60
						1	=	20

AVOIRDUPOIS WEIGHT.

lb.		oz.		drms.		grains.
1	=	16	=	256	=	7000
		1	=	16	=	437·5
				1	=	27·343

IMPERIAL MEASURE.

gallon		pints		fluid oz.		fluid drms.
1	=	8	=	160	=	1280
		1	=	20	=	160
				1	=	8

1 gallon = 70,000 grains of water at 16·7° C.
1 fluid ounce = $\frac{1}{20}$ pint = 437·5 ,, ,, ,,
1 gallon = 277·280 cubic inches.
1 fluid ounce = 1·733 ,,

FRENCH WEIGHTS AND MEASURES.

MEASURES OF LENGTH. ENGLISH.

	metre.		inches.		mile.	furlong.	yards.	feet.	inches.
Millimetre =	0·001	=	·03937	=	.	.	.	.	·03937
Centimetre =	0·01	=	·39371	=	..	..	..	..	·39371
Decimetre =	0·1	=	3·93708	=	.	.	.	.	3·9371
Metre =	1·0	=	39·37079	=	..	..	..	3	3·371
Decametre =	10·0	=	393·70790	=	.	.	10	2	9·7
Hectometre=	100·0	=	3937·07900	=	..	..	109	1	1
Kilometre =	1000·0	=	39370·79000	=	.	4	218	4	10·2
Myriometre=	10000·0	=	393707·90000	=	6	1	156	0	6

1 inch = ·0254 metre.
1 foot = ·3048 ,,

MEASURES OF CAPACITY.

1 litre = 1 cubic decimetre.

		litre.		cubic inches.		pints.
Millilitre, or Cubic centimetre(c.c.)	=	·001	=	·06103	=	0·00176
Centilitre	=	·01	=	·61027	=	0·01761
Decilitre	=	·1	=	6·1027	=	0·17608
Litre	=	1·0	=	61·027	=	1·76077
Decalitre	=	10·0	=	610·27	=	17·60778
Hectolitre	=	100·0	=	6102·7	=	176·07734
Kilolitre	=	1000·0	=	61027·0	=	1760·77341
Myriolitre	=	10000·0	=	610270·0	=	17607·73414

1 cubic inch = ·01639 litre.
1 cubic foot = 28·31531 litres.
1 gallon = 4·54336 ,,

MEASURES OF WEIGHT.

1 gram = the weight of 1 cubic centimetre (c.c.) of water at 4° C.

		grams.		grains.	Avoirdupois.			
Milligram	=	·001	=	0·01543				
Centigram	=	·01	=	0·15432				
Decigram	=	·1	=	1·54323				
Gram	=	1·0	=	15·43235		lbs.	oz.	drms.
Decagram	=	10·0	=	154·32349	=	0	0	5·65
Hectogram	=	100·0	=	1543·23488	=	0	3	8·5
Kilogram	=	1000·0	=	15432·34880	=	2	3	5
Myriogram	=	10000·0	=	154323·48800	=	22	1	2

1 grain = 0·0649 gram.
1 oz. (Troy) = 31·1035 grams.
1 lb. (Avoirdupois) = 453·593 ,,

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